

INDENTATION SIZE EFFECT IN WC-Co

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Declaration

I declare that this dissertation is my own, unaided work. It is being submitted for the degree of Doctor of Philosophy in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination in any other university.

(Signature of candidate)

_____ day of _____ - 2007

To Time...

....to well wishers and teachers...

Their teachings will be cherished for their skill, integrity, intelligence, singularity

This thesis is dedicated to Meenal Shrivastava and our parents & family

Abstract

Four different grades of WC-Co cemented carbides with constant average grain size and varying cobalt content were indented with a Vickers micro-indenter at loads ≤ 1 kg. The residual microindents were analysed by atomic force microscopy (AFM). The AFM images of the residual indents were analysed using the program AutoIndent specifically written for this work. Some of the main AutoIndent results are: the diagonals measured by AFM correlate with optical or scanning electron microscopy measurements if pile-ups of the material around the indent are included; the ratio of the diagonal to depth is not the same at all loads; the volume of the residual indent is not equal to the volume of the pile-ups of the material around the indent; and a change in shape of the pile-ups of material is observed when going from low cobalt content to high cobalt content. The results obtained from the AutoIndent program have been applied to the theory developed by Nix and Gao [Nix and Gao, 1998] of the size effect in microindentation. The Nix and Gao model is of the characteristic form $H/H_0 = \sqrt{1 + h^*/h}$, where H is the hardness for a given depth of indentation, h , H_0 is the hardness at the limit of infinite depth and h^* is a characteristic length that depends on the shape of the indenter, the shear modulus and H_0 . The characteristic length h^* is not a constant for a given material, but depends on the statistically stored dislocation density through H_0 . When rearranging the Nix and Gao equation to the form $H^2 = H_0^2 + h^*/h$, cemented carbides exhibit a “bilinear behaviour”, i.e. H^2 increases linearly with $1/h$ but the rate of increase is different for low and high $1/h$ values. The two lines cross over at a $1/h$ value equal to the reciprocal of the sum of the grain size of WC and mean free path of the Co binder. When expressing the hardness as a function of $\zeta = \lambda + \chi$, (where λ is the average WC grain size and χ is the mean free path of Co binder), $H = L/(\kappa^2 \zeta^2)$, it is found that, for a given load (L) the value of κ is

constant for the four grades of WC-Co tested. This work has demonstrated that (i) AutoIndent program allows for detailed characterization of the indents. (ii) The Nix and Gao model applied to WC-Co shows a bilinear behaviour of the material instead of the mono linear behaviour shown when applied to one-phase material [Nix and Gao, 1998]. (iii) The sum of the WC grain size and mean free path in Co is a characteristic length of the material controlling its plastic behaviour.

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Chapter 1

Introduction

It has been observed in most materials that the hardness measured by means of indentations increases as the indenting load decreases. This phenomenon is referred to as the indentation size effect (ISE). Although ISE was first reportedly observed by Griffith in 1920 [Griffith, 1921], the last two decades have seen a renewed interest in the phenomenon of ISE due to a renewed interest in indentations as tools to determine the mechanical properties of a material on a nano, sub-micron and micro scale (e.g. Stelmashenko *et al.*, 1993; DeGuzman *et al.*, 1993; Ma and Clarke, 1995; Poole *et al.*, 1996; McElhaney *et al.*, 1998; Lim and Chaudhri, 1999; Elmustafa and Stone, 2002, 2003; Swadener *et al.*, 2002a, 2002b, Oliver *et al.* 1986, Pharr *et al.* 1990).

Although the indentation size effect is a well documented phenomenon, attempts to explain the origin of the ISE or to model the ISE have had limited success. These attempts can be broadly categorized into two groups; (a) Corrections (or modifications) of the mathematical expression for hardness (b) Analysis of the physical processes occurring during indentation.

Maximum strain gradient (MSG) plasticity [Nix and Gao, 1998] and indentation (*elastic*) recovery [Luyckx et al., 1999] are two of the mechanisms that were considered in this work for explaining the behaviour or origin of ISE. Luyckx, Demanet and Shrivastava [Luyckx et al., 1999] have presented the first results on the effect of relaxation on the angles measured along the opposite edges and opposite faces of the residual indent, based on the AFM measurement of the residual indent in single crystal tungsten carbide. The correlation between the measured results and the calculated value of relaxed angle using plasticity characteristics of hardness measurements [Milman et al., 1993], [Galanov et al., 1999] is given in

Appendix G.

Nix and Gao [Nix and Gao, 1998] have developed the maximum strain gradient (MSG) theory by incorporating into the strain gradient plasticity theory [Ashby, 1970] geometrically necessary dislocations and statistically stored dislocations. Their MSG model has the form $H^2 = H_0^2 + h^*/h$, (referred to as Nix and Gao (NG) model or equation) where H is the measured hardness for the depth h of indentation, H_0 is the hardness without strain gradient effects (i.e. for large indentation depth) and h^* is a characteristic length that depends on the shape of the indenter, the shear modulus, and the Burgers vector (when the model is applied to one phase materials). For one-phase materials, the characteristic length scale observed experimentally for single crystal and polycrystalline material (Cu and Ag) correlates with theoretical values in the micrometer depth regime [Nabarro et al., 2006].

The NG model yields characteristic lengths of the order of micrometer scale for one phase material. An extension of the NG model to a two-phase material is explored herein. In this work the two-phase material investigated is tungsten carbide - cobalt (WC-Co), with an average WC grain size of $1.0 - 1.2\mu\text{m}$ and of varying cobalt content. The effect was investigated on a micrometer scale.

Broadly, three points are investigated, (i) to verify that WC-Co exhibits ISE. (ii) extend Nix and Gao's model to two-phase materials, since prior to this work application of the NG model to a two-phase material has not been reported; (iii) try to establish an empirical relationship between macrohardness and microhardness, since ISE prevents comparisons between the hardness of materials measured at low and high loads.

The first step in studying ISE is to analyze the residual indentations. A critical analysis of the residual indents plays a vital role in quantifying and understanding the ISE. For such purpose atomic force microscopy (AFM) was used because of its high spatial resolution capable of quantitative measurements. The residual indents geometrical information is then applied to ISE models. Finally the Vickers indenter geometry is compared to the information from AFM

This thesis is divided into seven chapters and eight appendices. In Chapter Two literature concerning indentation and ISE is reviewed. The experimental procedure is outlined in Chapter Three, while Chapter Four describes the method developed

to analyze AFM images of indentation. The results of indentation measurements are given in Chapter Five. In Chapter Six, Nix and Gao model is applied to the results. Finally, the conclusions and suggestions for future work are given in Chapter Seven.

Appendices deal with - (i) Finite element analyses: deformation an indenter undergoes while it is embedded into the samples under load. (ii) What defines the length of the diagonals, as compared by different techniques. (iii) Confocal stereology image of residual indent on a diffraction grating. (iv) AutoIndent program written ad hoc in Matlab for AFM images of residual indent. (v) Elastic recovery Theoretical indenter vs. experimental measured residual indent. (vi) An empirical expression relating the macro-and micro-hardness. (vii) Plasticity characteristic of hardness measurements (viii) Published papers relevant to his work are attached (a) Automated analysis of Vickers's Microindentations by Atomic Force Microscopy. (b) The size effects in micro indentation. (c) Elastic recovery in WC-single crystal.

Chapter 2

Literature Review

The indentation size effect (ISE) is the phenomenon where the hardness of the material appears to increase with decreasing indenting load. Although ISE was first reported (observed) by Griffith in 1921 [Griffith, 1921], the last two decades have seen a renewed interest in indentations as a tool for determining mechanical properties at a nano, sub-micron and micro scale.

Indentations are effectively explained on the macro-scale by classical-continuum models based on the cumulative work of many authors: Hill [Hill, 1950], Tabor [Tabor, 1951], Samuels [Samuels and Mulhearn, 1957], Mulhearn [Mulhearn, 1959], Sneddon [Sneddon, 1946], Dugdale [Dugdale, 1953], Timoshenko [Timoshenko and Goodier, 1951], Hirst [Hirst and Howse, 1969], Johnson [Johnson, 1970], Bishop [Bishop et al., 1945], etc. These theories and models have been successful in explaining the indentations and the plastic flow of the material. Finite element analysis (FEA) has been effective in this.

However the physical explanation of ISE within the framework of classical/continuum plasticity has been unsatisfactory. Experiments and theories are developing in attempting to explain ISE with a fair success in their prediction and simulations. A brief review of the prominent model of Nix and Gao [Nix and Gao, 1998] based on the strain gradient plasticity theory [Ashby, 1970] is described below and has been applied to this work.

Some of the physical mechanisms proposed for the explanation of the ISE with limited success in predicting ISE are: surface dislocation pinning [Varchenya et al., 1970], surface energy effects [Bernhardt, 1941], [Fröhlich et al., 1977], internal surface induced defects

[Hirao and Tomozawa, 1987], [Kokura et al., 1989], dislocation nucleation [Gane and Cox, 1970], work hardening during indentation [Mott, 1956], plastic deformation band spacing [Bull et al., 1989], indentation elastic recovery [Tate, 1945], ‘Rotating block’ model is based on the plastic relaxation by secondary dislocations [Brown, 1997], statistical size effects [Yost, 1983].

2.1 Indentation Size Effect

The origin of the ISE or to model the ISE can be broadly categorized into two groups; (a) Corrections (or modifications) of the mathematical expression for hardness, e.g PRS model. (b) Analysis of the physical processes occurring during indentation with a length scale eg. maximum strain gradient plasticity and without a length scale eg. elastic recovery.

Most of the models proposed for ISE are based on the power law or Meyer’s law [Meyer, 1908]. Meyer’s law is given by $L = ad^n$, where L is the indenting load, d is the diagonal of the resulting indentation impression, and the coefficient a and the exponent n vary with the indenter geometry. The coefficients in these models are justified by correlating them to some physical factors [Hays and Kendall, 1973], [Sargent, 1989], [Li and Brandt, 1992], [Gong et al., 1999] e.g. elastic properties of material, friction between indenter and specimen, energy-balance during loading-unloading cycle, etc.

The Proportional specimen resistance (PRS) model is a modification of the Meyers’ law $L = ad^n$, by introducing a linear term and/or including a volume term [Gong et al., 1998a] to the load-diagonal relationship. The physical meaning of the coefficient a and exponent n are not well explained or predicted.

The PRS models proposed by Hays and Kendall [1973] and Li and Brandt [1992] assume that as the load L is applied to the specimen it is partially affected by the material resistance L_r , and that the plastic flow is directly proportional to the indentation size d . The applied load and the indentation size are then related by

$$L - L_r = a_2 d^2$$

where $L_r = a_1 d$ gives the equation

$$L = a_1 d + a_2 d^2 \tag{2.1}$$

where a_1 is a constant related to the proportional resistance of the test specimen and a_2 is a constant related to the load-independent hardness or ‘true hardness’. The term $a_1 d$ has been related to the energy consumed in creating new surface as indentation facets and microcracking [Fröhlich et al., 1977], and the same term has been related to frictional and elastic contributions to PSR model by [Li and Bradt, 1993]. The term $a_2 d^2$ was thought to be the work of permanent deformation [Fröhlich et al., 1977] or the volume energy of deformation [Li and Bradt, 1993].

PRS model has been extensively applied by [Sangwal et al., 2002] to a range of materials, by rearranging equation 2.1 between the measured variables load and diagonal a linear correlation was observed. Despite this, the model fails to provide an insight into the behaviour of the material or predict the indentation size effect of the material or explain its origin.

A theory incorporating geometrically necessary dislocations in the strain gradient plasticity theory appears to explain the ISE. A brief review of the experiments and theories leading up to the formulation of the Nix and Gao model is mentioned below.

The observation of the variation of hardness at lower loads by Gane and Cox [Gane and Cox, 1970] has suggested, that in very small deformed volumes it may not be possible to accumulate a sufficient number of dislocations to give rise to an appreciable increase in flow stress. They show that work hardening does not occur at small indentation sizes suggesting that the work hardening process in small volumes is different than in the bulk. They conclude that, the increase in flow stress produced by bulk work hardening is still effective at a small size. Thus, the increase in hardness with decreasing size is as a result of an increased stress required to generate dislocation sources at small sizes.

Ashby [Ashby, 1970] in discussing the deformation of "plastically non-homogeneous material" introduced the concept of gradients of plastic deformation, stating that the geometrically necessary dislocations (GNDs) density (ρ_G) controls the work hardening of the specimen when their density exceeds the statistically stored dislocations (SSDs) density (ρ_S). The density of GNDs is directly related to the gradient of plastic deformation which varies inversely to the length over which the deformation takes place. The geometrically necessary dislocations density (ρ_G)

is given by

$$\rho_G = \frac{4\gamma}{\ell b} \quad (2.2)$$

where b is the Burgers vector, γ is the shear strain and ℓ is the length scale. Two-phase alloys work-harden much faster than pure single crystals, such alloys are “plastically non-homogeneous” as gradients of plastic deformation are imposed by the microstructure.

The flow-stress law containing the strain-gradient term was proposed by Aifantis [Aifantis, 1984] for the equivalent flow stress containing a material internal length scale. This concept was further developed by Fleck *et al.* in their work on “The phenomenological theory of strain gradient plasticity” (SGP) [Fleck et al., 1993], with experimental support to the theory [Fleck et al., 1994], and further advanced by incorporating elastic and plastic strains in the formalization of the SGP theory [Fleck et al., 2001].

The strain gradient plasticity theory and the geometrically necessary dislocations have been combined with the Taylor-based nonlocal theory of plasticity [Gao and Huang, 2001], which have been applied to theory and to experimental data by [DeGuzman et al., 1993], [Stelmashenko et al., 1993], [Fleck et al., 1994], [Ma and Clarke, 1995], [Nix and Gao, 1998], [Gao et al., 1999a], [Gao et al., 1999b], [Huang et al., 1999], [Huang et al., 2000], [Gao and Huang, 2001], [Nix, 2002], [Huang et al., 2006] leading to the micromechanical basis of size-dependent plasticity models accounting for GNDs.

Within the context of the SGP, GND plays a key role in the various models proposed for ISE. Needleman and Gil Sevillano [Needleman and J. Gil Sevillano, 2003] in their review on “Viewpoint set on: geometrically necessary dislocations and size dependent plasticity”, along with other authors have heaved caution towards understanding of these (GNDs & SGP), and independently Mughrabi [Mughrabi, 2004] as well. In spite of this, the model proposed by Nix and Gao (NG model) [Nix and Gao, 1998] $H = H_0 \sqrt{1 + h^*/h}$, where H and h are the measured hardness and residual depth of the indent, fits most of the reported ISE. Variation of NG models includes an additional (or correction to) hardness term i.e. $H - H_1$, where H_1 is the work

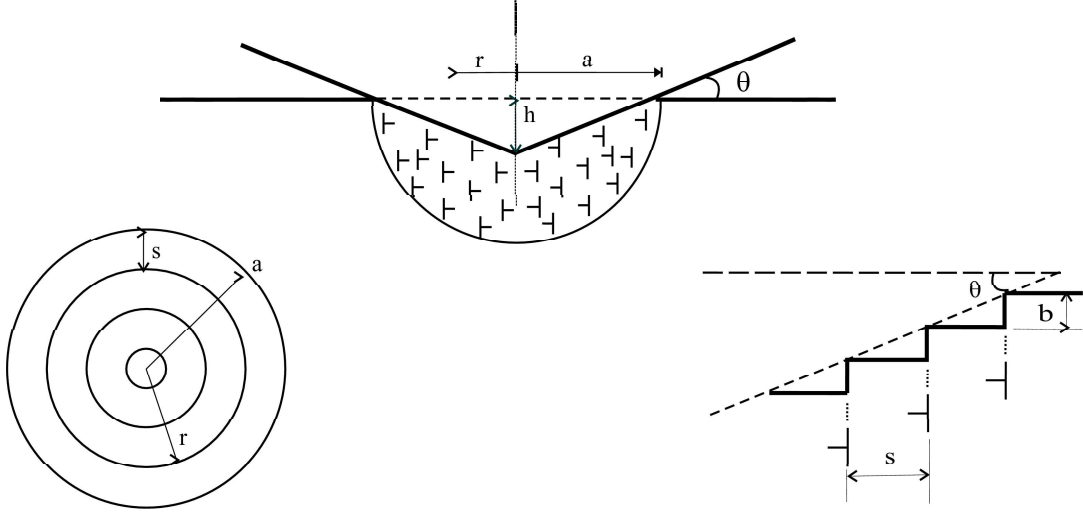


Figure 2.1: The indentation model of De Guzman [DeGuzman et al., 1993], Nix and Gao [Nix and Gao, 1998]. Indentation by a rigid circular cone proceeds by the glide of circular edge dislocations normal to the surface.

hardening component representing the increase in hardness from the onset of yielding to an effective strain factor [Swadener et al., 2002], or H_1 is the hardness due to friction stress [Elmustafa et al., 2004]. In this work we will only be dealing with the NG model as developed in Nix and Gao [1998].

2.2 Maximum strain gradient plasticity model

The Maximum strain gradient plasticity model was first analysed by De Guzman *et al.* [DeGuzman et al., 1993], and developed in detail by Nix and Gao [Nix and Gao, 1998], [Gao et al., 1999b], [Gao et al., 1999a], [Huang et al., 1999], [Huang et al., 2000]. Essentially the same results, with somewhat different numerical coefficients, had previously been published without derivation by Stelmashenko *et al.* [Stelmashenko et al., 1993]. In this work the details of Nix and Gao are followed, as in Nabarro *et al.* [Nabarro et al., 2006].

The indenter is assumed to be a rigid circular cone of half-angle θ (figure 2.1).

The diameter of the indentation is $2a$, its depth h . Hence

$$h = a \tan \theta. \quad (2.3)$$

In the case of a homogeneous deformation, the critical resolved shear stress τ on a glide plane is given in terms of the dislocation density ρ_s by

$$\tau = \alpha \mu b \rho_s^{1/2}, \quad (2.4)$$

where α is taken to be $1/2$, b is Burgers vector and μ is the shear modulus [Taylor, 1934].

Here, ρ_s is the density of “statistically stored” dislocations. When the deformation is inhomogeneous, involving either a strain gradient or a curvature k (of dimensions of L^{-1}), there is also a density ρ_g of geometrically necessary dislocations (GNDs) given in order of magnitude by

$$\rho_g = k/b. \quad (2.5)$$

The value of ρ_s at any point in a small indentation is taken to be the dislocation density at the geometrically corresponding point in a large indentation. It is assumed that the critical resolved shear stress is then given by

$$\tau = \alpha \mu b (\rho_s + \rho_g)^{1/2}. \quad (2.6)$$

For a polycrystal, the tensile flow stress σ is given by the von Mises relation [Cottrell, 1967] to be

$$\sigma = \sqrt{3} \tau. \quad (2.7)$$

The hardness H is given by

$$H = 3\sigma = 3\sqrt{3}\tau = 3\sqrt{3}\alpha\mu b(\rho_s + \rho_g)^{1/2}. \quad (2.8)$$

The hardness of a large indentation is

$$H_0 = 3\sqrt{3}\alpha\mu b(\rho_s)^{1/2}. \quad (2.9)$$

In estimating ρ_g , it is assumed as in figure 2.1 that the plastic deformation during indentation proceeds by simple prismatic punching, loops of dislocations being equally spaced along the surface of contact. Then the spacing s is given by

$$s = b \cot \theta. \quad (2.10)$$

The length of a dislocation loop of a radius r is $2\pi r$, and the number of loops between r and $r + dr$ is dr/s . The total length λ of dislocation line is thus

$$\lambda = \int_0^a \frac{2\pi r dr}{b \cot \theta} = \frac{\pi a^2 \tan \theta}{b}. \quad (2.11)$$

The plastically deformed region is assumed to be a hemisphere of radius a and volume

$$V = \frac{2}{3}\pi a^3. \quad (2.12)$$

The density of geometrically necessary dislocations is thus

$$\rho_g = \frac{\lambda}{V} = \frac{3 \tan^2 \theta}{2bh}. \quad (2.13)$$

It follows from 2.8, 2.9 and 2.13 that

$$\left(\frac{H}{H_0} \right)^2 = 1 + \frac{h^*}{h}, \quad (2.14)$$

where

$$h^* = \frac{81}{2} b \alpha^2 \tan^2 \theta \left(\frac{\mu}{H_0} \right)^2. \quad (2.15)$$

Nix and Gao showed that the experimental observations both on the (111) face of a single crystal of copper and on cold-worked polycrystalline copper gave remarkably accurate linear relations between $(H/H_0)^2$ and $1/h$. They noted that h^* is not a constant for a given material and indenter geometry, rather it depends on the statistically stored dislocation density through H_0 .

If figure 2.1 is taken to represent indentation by a wedge rather than by a circular cone, the length of geometrically necessary dislocation line per unit length of indentation [DeGuzman et al., 1993] is

$$L = \int_0^a \frac{2dr}{b \cot \theta} = \frac{2a \tan \theta}{b}, \quad (2.16)$$

while the plastically disturbed area is

$$A = \frac{1}{2}\pi a^2, \quad (2.17)$$

leading to a geometrically necessary dislocation density of

$$\rho_g = \frac{L}{A} = \frac{4 \tan^2 \theta}{\pi bh}, \quad (2.18)$$

differing little from equation 2.13.

2.2.1 Some assumptions of the model

Equation 2.6 is an approximation as pointed out by [Nabarro et al., 2006]. The dislocation density tensors for statistical and for geometrically necessary dislocations are not isotropic, but display the geometry of the strain and of the strain gradient respectively. Their interaction depends on the slip systems actually available (e.g. in fcc or bcc structures).

The prismatic dislocation loops of figure 2.1 represent the most efficient way of meeting the geometrically necessary dislocation requirements. Dislocations of the necessary glide system will generally not be available and the actual density of geometrically necessary dislocations will exceed that given by 2.11 by a factor of order $\sqrt{3}$.

It is assumed in figure 2.1 that the plastically deformed region is a hemisphere of radius a . The observations of Dyer [Dyer, 1961] for the indentation of the (100) face of a single crystal of copper by a sphere show heavy concentration of dislocations in a region which is roughly a sphere of radius $3a$, so that V would exceed that given by 2.12 by the large factor of 54. The survey by Samuels [Samuels, 1986] shows that the geometry of the heavily deformed region depends on the form of the stress-strain curve, specifically for an ideal elastic-plastic model on the value of $(E/Y) \cot \theta$, where E is Young's modulus and Y the yield stress.

Motz *et al.* [Motz et al., 2004] have measured the local crystallographic orientation change due to the indentation under the indenter flank in copper grains, showing the misorientation distribution to be extended up to $2a$. The orientation gradient associated with the strain gradient caused by GNDs is $1.8^\circ/\mu\text{m}$ or $31.4\mu\text{m}$ per radian. Durst *et al.* [Durst et al., 2005] have considered the plastically deformed volume underneath the indenter by approximating the size of the plastic zone at an equivalent plastic strain of $\approx 1.5\%$, using the contact radius to be $fa = 1.9a$; the density of GNDs is then $1/f^3$ of the value (10).

These two considerations suggest that ρ_g is usually about $\sqrt{3}/10$ times the value given by Eq. 2.13, and h^* correspondingly smaller by a factor of about 0.67.

The model implicitly assumes that the statistically stored and geometrically necessary dislocations are distributed similarly in space. This may well not be true. However, the consideration is not relevant in the present approximation

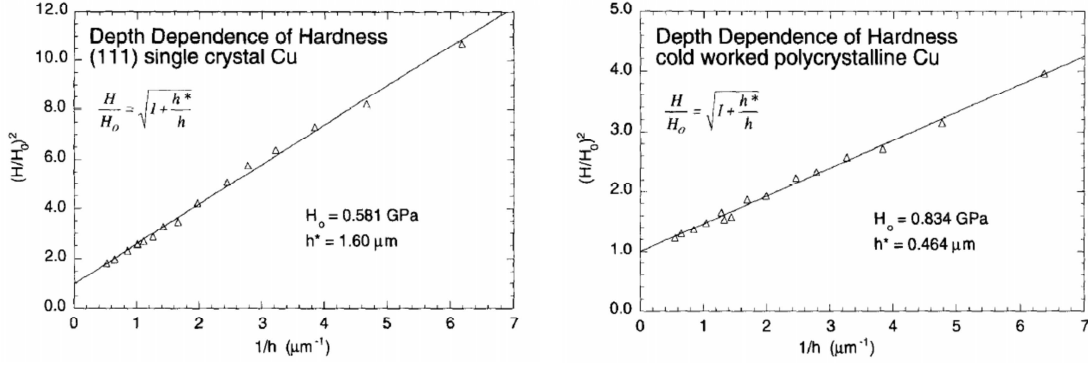


Figure 2.2: Depth dependence of the hardness of (111) single crystal copper and cold worked polycrystalline copper (Figure 3 and 4 respectively of Nix and Gao [Nix and Gao, 1998]).

which considers only mean dislocation densities and adds them linearly.

Stilwell and Tabor [Stilwell and Tabor, 1962] showed that, when a conical indenter is removed from an indentation in steel or a commercial alloy, the recovery in depth is almost purely elastic. Nevertheless, the loops of dislocation in figure 2.1 represent a dislocation pile-up which has resisted the initial plastic indentation. This kinematic hardening implies that the increase in hardness produced in a small indentation by the geometrically necessary dislocations is greater than that implied by equation 2.6, and the value of h^* correspondingly larger. This partially compensates the reduction in h^* estimated from geometrical considerations.

2.3 Extension of the model

Equation 2.14 predicts that the plot of $(H/H_0)^2$ against $1/h$ will be a straight line, but does not predict the slope of the line. If Eq. 2.14 is rewritten as

$$H^2 = H_0^2 + \frac{81}{2} \alpha^2 b \mu^2 \tan^2 \theta \frac{1}{h}, \quad (2.19)$$

the slope of the plot of H^2 against $1/h$ is determined by the numerical factor $81\alpha^2/3$, the material constant $b\mu^2$ and the geometrical factor $\tan^2 \theta$ of the indenter. This provides a quantitative test of the model. Nix and Gao assumed $\alpha = \frac{1}{2}$; the usual value is $\alpha = 1/3$ [Tabor, 1951]. The experimental slopes shown for copper in figure 2.2 of Nix and Gao are compared in table 2.1 with those predicted by

Table 2.1: Comparison of predicted and measured values of the characteristic length h^* for values of $\alpha = \frac{1}{2}$ and $\frac{1}{3}$.

Material	H_0 (GPa)	h^* (μm)	μ (GPa)	b (nm)	α	h^* (predicted) (μm)
(111) Single crystal Cu (annealed)	0.581	1.60	42	0.256	1/2	1.73
	0.581	1.60	42	0.256	1/3	0.77
Polycrystalline Cu (cold worked)	0.834	0.464	42	0.256	1/2	0.840
	0.834	0.464	42	0.256	1/3	0.373

Eq. 2.15 with the values $b = 2,56 \times 10^{-10}\text{m}$, $\mu = 42$ GPa, $\tan \theta = 0.358$ and $\alpha = \frac{1}{2}$ or $\alpha = \frac{1}{3}$. The agreement is satisfactory.

The analysis by Fleck *et al.* [Fleck et al., 1994] of torsion experiments on cold-drawn copper wires led to values of h^* between 2.6 and $5.1 \times 10^{-6}\text{m}$, with a mean of $3.7 \times 10^{-6}\text{m}$. The analysis by Stölken and Evans [Stolken and Evans, 1998] of bending tests on thin foils of polycrystalline copper led to values in the range $3 - 5 \times 10^{-6}\text{m}$.

A further check is possible. The experiments analysed by Nix and Gao were performed in a range of hardness centred around 1.3 GPa, corresponding to a resolved shear stress of $1.3/3\sqrt{3} = 0.25$ GPa. The survey by Basinski and Basinski [Basinski and Basinski, 1979] of dislocation densities in single crystals of copper deformed in tension shows a corresponding dislocation density of about 10^{-12}m^{-2} , with a separation between statistically stored dislocations of $1 \times 10^{-6}\text{m}$. In the present theory, h^* is the reciprocal of the strain gradient at which the separations of neighbouring geometrically necessary dislocations and of neighbouring statistically stored dislocations are equal. The agreement is again satisfactory.

The existence of a characteristic length of the order of a micron in copper strained by about 10 percent seems well established. The length has no significance in the perfect copper crystal; it appears only on plastic deformation. It arises from a balance between the dislocation densities associated with homogeneous plastic strain and with plastic strain gradient.

2.4 Direct observation of the characteristic Length

Although the existence of a characteristic length in the plastic deformation of copper has been demonstrated, its value has been derived from an analysis of mechanical measurements. A direct observation has recently been published by Motz *et al.* [Motz et al., 2004], Lloyd *et al.* [Lloyd et al., 2005]. It is first necessary to recall that a component of strain such as $e_{ij} = (\partial u_j / \partial x_i + \partial u_i / \partial x_j)$ and a component of rotation such as $\omega_{ij} = \frac{1}{2}(\partial u_j / \partial x_i - \partial u_i / \partial x_j)$ are both linear combinations of components of the same displacement gradient tensor, and that their gradients such as $\partial e_{ij} / \partial x_k$ and $\partial \omega_{ij} / \partial x_k$ are quantities of the same kind. Motz *et al.* [Motz et al., 2004] made Vickers indentations of diameters 3.3 – 16.3 microns in grains of polycrystalline copper of mean grain size 150 microns, thus effectively single crystals of random orientation. The indentations were sectioned normal to the surface, and the lattice rotations were measured by electron-back-scattering diffraction. They found that the region of highest orientation changes are concentrated beneath the indent flanks and below the indent tip. The rotation patterns did not scale with the size of the indentation. Instead, the width of the regions of maximum misorientation were constant at about 1 micron, while the ratio of the maximum misorientation to the width of the Vickers imprint was constant at about 1.8 degrees per micron. This corresponds to a radian in 31.8 microns of indentation diameters. The principal rotation gradient is in depth, and the gradient in depth is a radian in 4.5 microns, larger than but of the same order as other estimates of h^* [Nix and Gao, 1998], [Fleck et al., 1994], [Stolken and Evans, 1998].

Lloyd *et al.* [Lloyd et al., 2005] have used a similar technique examining nanoin-dents and the deformation behaviour in a range of single crystal materials with differing resistances to dislocation flow, phase transformation (silicon and germanium), twinning (gallium arsenide and germanium at 400°C), lattice rotations (spinel MgAl_2O_4), shear (spinel), lattice rotations (copper) and lattice rotations and densification (TiN/NbN multilayers). The magnitude of the lattice rotation were measured at different positions under the indents. All the samples show varying degree of lattice rotation under the indent. But no rotation were observed to the side of the indent i.e. parallel to the surface. In the case of copper, the results

for the lattice rotation were similar to those observed by Motz [Motz et al., 2004].

2.5 Bilinear behaviour

Elmustafa *et al.* [Elmustafa and Stone, 2002], [Elmustafa et al., 2004] have reported “bilinear” behaviour for the ISE in polycrystalline high-purity fcc aluminum and α -brass by combining microindentation and nanoindentation. The residual indent area (A) measured by optical (OM) and scanning electron (SEM) microscopes and the area calculated based on contact stiffness taking into account machine compliance shows a linear correlation between the $\sqrt{A}$ measured by these three techniques by a factor of 1.03. Of interest is that a correlation of ≈ 1 is observed only when the pileups are included in the OM and SEM measurements. However no explanation or justification are given for the inclusion of the pileups around the material.

Elmustafa *et al.* have modified the NG model to take into account the decrease in hardness due to friction H_f . The “corrected” hardness $H - H_f$ is given by

$$\frac{(H - H_f)^2}{4\Gamma\zeta^2\alpha^2\mu^2b} = \frac{b\rho^s}{4\Gamma} + \frac{1}{D}, \quad (2.20)$$

and the density of the dislocations is $\rho_G = 4\Gamma/b\Omega D$, where Γ is $\simeq 19\%$ that is the shear strain in the plastic zone beneath the indenter, while ΩD is a characteristic length over which the strain gradient exists, $\Omega \simeq 1.0 - 1.2 \text{ m}^{-1}$, $\zeta = 9$ and $\alpha = 1/3$.

Although D has not been explicitly defined as the size of the indent it is taken to be the depth of indentation. A plot of $\frac{(H-H_f)^2}{4\Gamma\zeta^2\alpha^2\mu^2b}$ vs. $\frac{1}{D}$ was given, where D is substituted by $D = \sqrt{A}$. Such a plot for the combined micro- and nano-indentation reveals a "bilinear behavior" as shown in figure 2.3.

A critical flaw in the representation of the experimental data (figure 2.3) using the above equations 2.20 is in particular due to $D = \sqrt{A}$. When using $1/D = 1/\sqrt{A}$ for microindentation $\sqrt{A} = D = 0.7d$, where d is the diagonal of the residual Vickers indent, while for nanoindentation $\sqrt{A} \approx 5h_c$, where h_c is the contact depth between the indenter and the material.

The nanoindentation area is based on contact depth, thus, this assume that there is no relaxation of the material during unloading. Similarly, in case of the Vickers microhardness $\sqrt{A} \simeq 0.7d$ or $1/D = 1/\sqrt{A} = 1.3/d$, and $D \simeq 5h$ where h

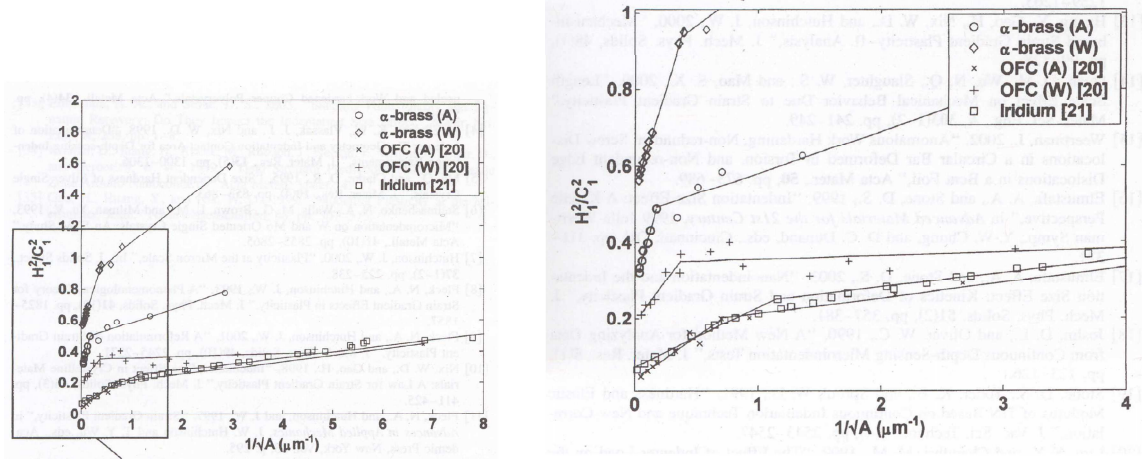


Figure 2.3: H_1/C_1 versus $1/\sqrt{A}$ for α -brass, Oxygen free copper (A - annealed mechanically polished; w- work hardened electropolished) and iridium samples, where $H_1 = H - H_f$ and $C_1 = 4\Gamma\zeta^2\alpha^2\mu^2b$. (Figure 5 from [Elmustafa et al., 2004] and reference there in.)

is the residual depth. While the ratio of d/h for the residual Vickers indent is > 7 . Both these cases assumes no relaxation of indentation depth and that the strain gradient from this point onward till the final residual depth is achieved remains unchanged.

The integrity of using the common $\sqrt{A}$ between the micro- and the nano-indentation data, depends on the conversion of the measured diagonal of the residual indents to the contact depth of nanoindentation the measured. This leads to a misconception that the characteristic length ΩD is related to the point when the indenter is in contact with the surface (under loading), while the strain gradient plasticity model deal with the point when the indenter is fully unloaded.

In order to maintain the same form of measurement of depth, a conservative conversion factor of $1/h \simeq 5 \times 1/\sqrt{A}$ ought to be applied. Then the gradient of the slope for the microhardness and the nanoindentation would become approximately the same. Hence, the bilinear behavior observed by Elmustafa *et al.* [Elmustafa et al., 2004] is an artifact due to the difference in diagonal and depth measurements, as well as the fact that the area is calculated when the indenter is still in full contact with the material.

2.6 Cemented Carbides

The structure of tungsten carbide-cobalt (WC-Co) alloys can be described in two ways. The carbide grains can be considered to form a rigid continuous skeleton cemented in the cobalt binder, or the carbide can be considered as a dense dispersoid in the binder, with a thin binder film separating the individual carbide grains. In the former case, the plastic deformation requires considerable plasticity in the carbide as the whole skeleton must deform [Ivensen et al., 1964] with increased dislocation density from 10^8 to $10^{10}/\text{cm}^2$, while in the later case deformation strain hardening of the binder occurs as a result of dislocation tangling upon deformation [Doi et al., 1972].

WC has a hexagonal crystallographic structure with two atoms (W and C) per unit cell as illustrated in figure 2.4. The lattice constants are $a = 2.906 \text{ \AA}$ and $c = 2.837 \text{ \AA}$ with $c/a = 0.976$. The carbon atoms are located either all at $\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$ or at $\frac{2}{3}, \frac{1}{3}, \frac{1}{2}$ lattice spaces in the simple hexagonal lattice of the tungsten atoms. Mechanically WC is a hard and brittle material and glides along the prismatic plane $\{1\bar{1}00\}$ in the $\langle 11\bar{2}3 \rangle$ direction [Luyckx, 1970]. The shortest Burgers vector having components on both $\langle 0001 \rangle$ and $\langle 2110 \rangle$ directions is the vector $\frac{1}{3}[2\bar{1}\bar{1}3]$ of $4.06 \text{ \AA}$.

Cobalt is hexagonal closed packed (hcp) crystal structure with lattice parameters $a = 2.507 \text{ \AA}$ and $c/a = 1.623$ at room temperature. It is stable as face-centred crystal (fcc) above 413°C [Bollmann, 1961], [Kamel and Halim, 1966].

A typical microstructure of WC-Co is given in figure 2.5, where the bright regions are the WC grains and the dark region is the Co binder matrix. The different shapes of the grain are indicative of heterogeneous orientation of the WC-grains. During indentation the deformation in WC-Co is due to plastic deformation of WC grains and of cobalt binder phase. The plastic deformation of WC-Co occurs primarily by;

1. Contacting carbide grains and thin areas of cobalt phase make the primary contribution to elastic deformation of WC-Co.
2. Plastic deformation by dislocation in WC grains
3. Dislocation interaction at WC/WC and WC/Co grain boundaries by glide

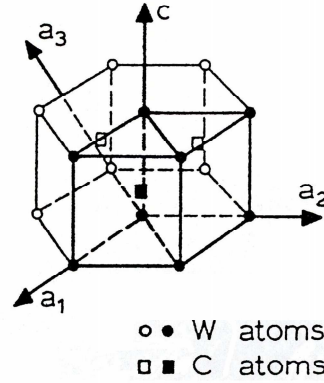


Figure 2.4: WC structure

and climb.

WC deformation

In WC grains the deformation mechanism for (0001) WC consists of slip lines which are produced by dislocations gliding on $\{01\bar{1}0\}$ planes along $\langle 2\bar{1}\bar{1}3 \rangle$ directions. [Luyckx, 1970], [Hagege et al., 1980], [Hibbs and Sinclair, 1981], [Hibbs et al., 1984], [Luyckx et al., 1992]. The carbide grains in as received material contain dislocations with a density in the range $10^8 - 10^{10}/\text{cm}^2$.

Co deformation

Although the cobalt phase in cemented carbide has mainly fcc structure, the structure changes to the hcp structure under stress [Suzuki et al., 1967]. This structural transformation could be due to the hcp structure being the equilibrium room temperature structure and that stress can supply the activation energy necessary for the movement of cobalt atoms. The orientation relationship between the phases in fcc $\rightarrow$ hcp transformation is most probably $(111)_{fcc} || (0001)_{hcp}$ $[110]_{fcc} || [11\bar{2}0]_{hcp}$ [Sarin and Johannesson, 1975] and transformation shear direction $\langle 10\bar{1}0 \rangle_{hcp} || \langle 112 \rangle_{fcc}$ [Nelson and Altstetter, 1964] The cobalt-rich binder, with tungsten and carbon in solid solution, contains a high density of stacking faults.

Models for hardness of cemented carbides (WC-Co) have been discussed by [Makhele-Lekala et al., 2001], [Engqvist et al., 2002] using the mechanism of the plastic deformation of WC-Co. Makhele *et al.* has discussed the plastic deformation

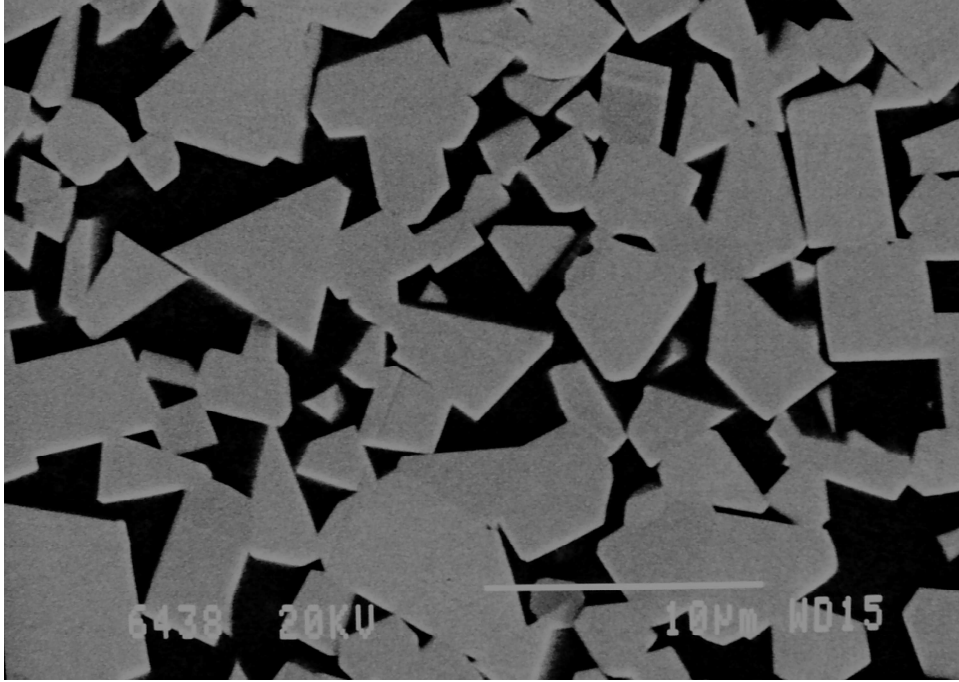


Figure 2.5: WC-Co microstructure.

of WC-Co on an atomistic level dealing with the theory of dislocations. In their model they assume a continuous carbide skeleton. In WC-Co, Co is the soft phase and the dislocations are easily formed. The dislocation movement is impeded by the WC grains, which act as an obstacle. The dislocations pile-up at the grain boundaries, deforming WC grains plastically. The hardness model proposed by Engqvist *et al.* is based on the physical interpretations of the microstructure of cemented carbides and deformation of thin binder layer and fine-grained materials.

ISE has been noticed by Jia *et al.* [Jia et al., 1998], who have observed that hardness decreases with increasing load in the low load region and varies little when the indenting load is greater than 10kg, in various grades of cemented carbides with nano size ($0.07\text{ }\mu\text{m}$), to fine ($0.8\text{ }\mu\text{m}$) and coarse ($> 1\text{ }\mu\text{m}$) WC grains. The variation of hardness with load is attributed to micro-cracking [Roebuck and Almond, 1988] and work-hardening in the stressed region and the work-hardening is relatively small at low deformation. Their results suggests that for finer grades of WC-Co a higher micro-fracture strength occurs within the region stressed by the indentation.

There are four types of micro-fracture process involved with the plastic deformation (i) cracking of the WC grains of the Co rich binder phase, at the WC/WC interfaces, and at WC/Co interface, cracks occurring beneath the surface of the stressed region and or minute “invisible” cracks.

Chapter 3

Experimental

3.1 Specimens

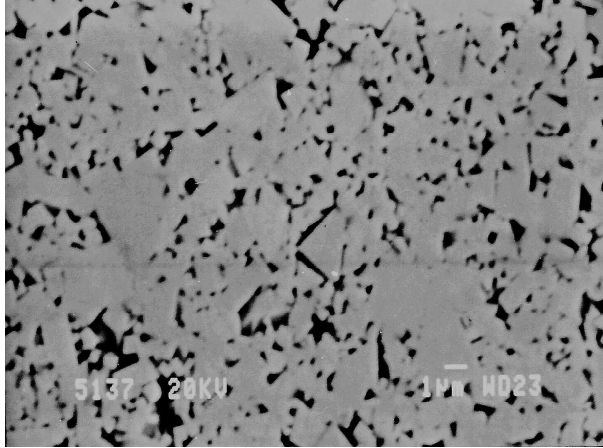
A tungsten carbide single crystal and four WC-Co samples with 6, 11, 15 and 20 wt% percent cobalt binder were used for this study of the indentation size effect. Figure 3.1 (page 22) shows SEM micrographs of the four samples. The hard phase, i.e. the WC grains had an average grain size of $1.0 - 1.2\mu\text{m}$. The samples were polished using diamond powder of particle size down to $0.25\mu\text{m}$. After polishing, they were cleaned ultrasonically immersed in alcohol.

The mechanical properties of WC-Co composites are strongly influenced by their microstructures. In a two-phase material of known composition, the metallographic parameters such as grain size and mean free path influence the mechanical performance and physical properties. In table 3.1 (page 21) some of the metallographic parameters and the hardness are given.

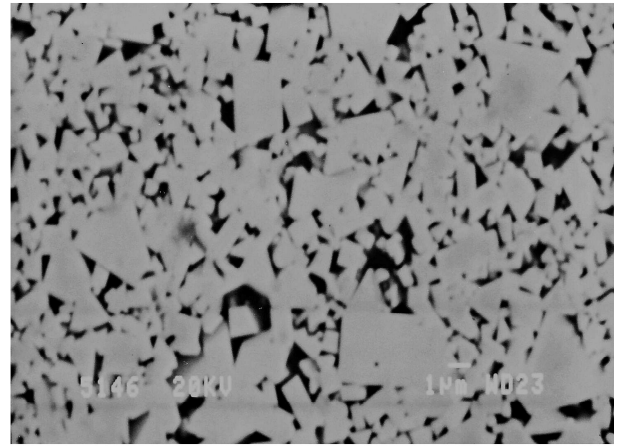
The mean free path (χ), grain size (λ) and the cobalt volume fraction (V_{Co}) obey the relationship $\chi = \lambda(0.2 - 0.7V_{Co} + 6V_{Co}^2)$ derived by Luyckx and Love [Luyckx and Love, 2006].

Table 3.1: Summary of the grain size of WC grains, mean free path of cobalt binder and the Vickers hardness for the four WC-Co samples.

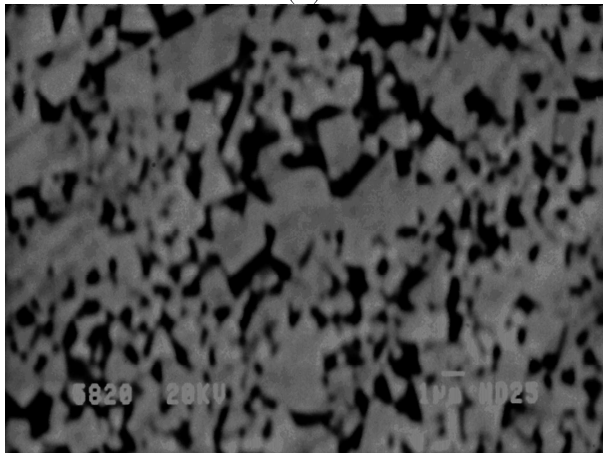
Sample Name	S6	S11	S15	S20
Cobalt wt.%	6	11	15	20
Cobalt Vol.% V_{Co}	10.1	17.8	23.7	30.5
WC mean grain size μm) (λ)	0.98	1.07	1.10	1.17
Mean free path (χ) μm)	0.33	0.54	0.75	1.07
Vickers Hardness (HV) (GPa)	17	15.5	11.7	10.7



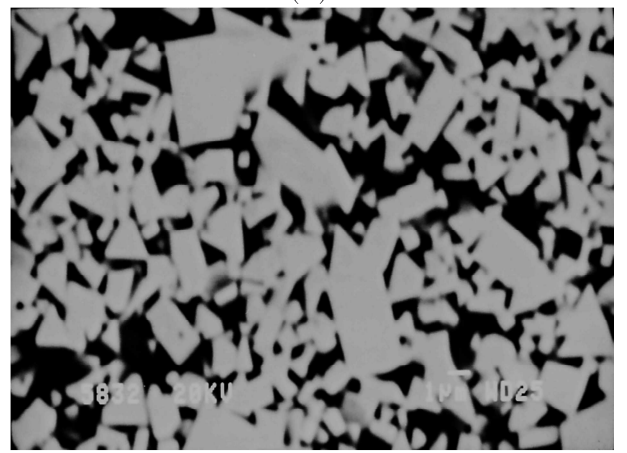
(a)



(b)



(c)



(d)

Figure 3.1: SEM micrographs showing the microstructure of (a) WC 6 wt.% Co, (b) WC 11wt.% Co, (c) WC 15 wt.% Co, (d) WC 20 wt.% Co with mean grain size $1 - 1.2\mu\text{m}$.

3.2 Indentations

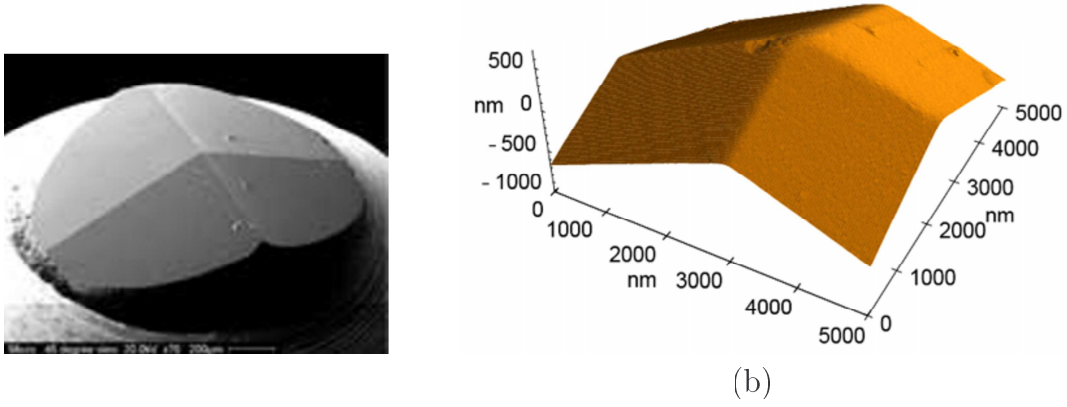


Figure 3.2: SEM and AFM image of the Vickers diamond indenter [Hasche et al., 1998].

The samples were indented using a Micro-Vickers hardness tester LECO M-400 in accordance with the guidelines ASTM E384¹. The applied indenting load (or test load) were: 0.05, 0.1, 0.2, 0.3, 0.5 and 1 kgs. Four indentations per test load were performed on each sample. The indentations at 1 kg load were assumed to give the macrohardness of the specimens. The spacings between successive indents were about 5 folds the diagonal of indent. The dwell time for the load was 20 seconds.

The Vicker's diamond indenter is of square-based pyramidal geometry with apex angle between opposite faces ("opening angle") of 136° , while the angle between the opposite edges is 148.11° . An image of the Vickers indenter is shown in figure 3.2.

3.2.1 Measurement of Residual Indents

The "residual" indent is the impression of the indenter after removal of the indenting load. The Vickers hardness (H), is mathematically defined as the ratio of the applied load (L) to the actual contact area (A) between the indenter and the sample [Smith and Sandland, 1925]. The contact area (A), is a function of indenter geometry and is calculated from the measured diagonal (d) as $A = d^2 / (2 \sin 68^\circ)$.

The diagonals of the residual indent were measured using optical microscopy (OM) and atomic force microscopy (AFM). For the samples WC 15 wt.% Co

¹ASTM E 384-99: Microindentation Hardness of Materials

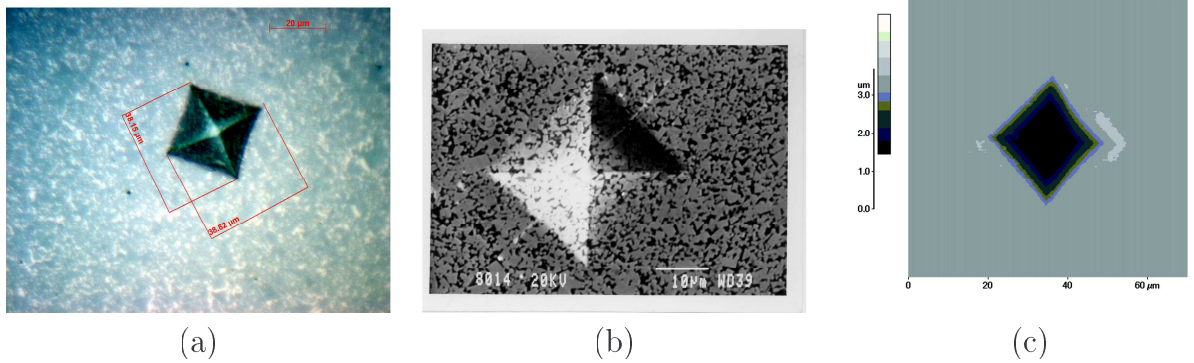


Figure 3.3: Residual indentation as observed by (a) optical microscopy (scale bar $20\ \mu\text{m}$ at the upper top corner of the image), (b) scanning electron microscopy (scale bar $10\ \mu\text{m}$ at the bottom right corner of the image) and (c) atomic force microscope (Scan Size $70\ \mu\text{m} \times 70\ \mu\text{m}$) for 1 kg indenting load on WC 15 wt.% Co.

the diagonals for all the indenting loads tested were measured by OM, scanning electron microscopy (SEM) and AFM. Figure 3.3 shows the residual indents for the applied load of 1 kg.

3.2.2 Optical Microscopy

In standard hardness testing the diagonals of the indents are measured using the pre-mounted optical microscope on the LECO M-400 tester at magnifications of X55 and X100. The diagonals are measured by focusing the OM, at a magnification at which the entire residual indent is within the field of view. Thereafter two markers are positioned at the opposite corners of the indent and readings are taken on a micrometer gauge attached to the tester. The markers are positioned at the dark-bright boundary, indicative of the corners of the residual indent (as shown in figure 3.3 (a)). The residual indent and the ‘original’ surface are represented by the dark ‘squarish’ region and the brighter region respectively.

Surface characterization by optical (or light) microscopy has been the subject of several research publications, reports, (manufactures’s application) bulletins [Miyoshi, 2002], [Latimer, 1979] evaluating resolution, resolvability, size of features, etc. Optical microscopy in its general form (i.e. excluding scatterometry,

interferometry or profilometry) has a variable lateral and vertical resolution depending on the size of the surface in the field of view and the wavelength of light used. For optical magnification of X1000 a lateral resolution of $\simeq 0.1\mu\text{m}$ is attainable while the depth of field (ability to clearly distinguish a surface feature or property as a function of depth) resolution is $\approx 0.2\mu\text{m}$.

In microindentation measurements once the entire indent is within the field of view, the diagonals are measured. For the cemented carbides tested, the diagonals are in range of 5 - 50 μm and at a magnification of X100 the lateral resolution is $\approx 0.22\mu\text{m}$. Although, several optical microscopies can attain vertical resolution of the order of nm (eg. differential interference contrast microscopy, optical profiler, laser interferometry, confocal microscopy, optical scatterometry) none of these techniques were applied to the microindentations under study as they all have similar lateral resolution of $\simeq 0.1\mu\text{m}$, hence, they would not have provided additional information.

3.2.3 Scanning Electron Microscopy

The microstructure of undeformed WC-Co and of the indentations for the S15 samples were observed by a scanning electron microscopy (SEM) in a JEOL JSM-840 in the secondary electron mode. The SEM micrographs of the polished undeformed surface for all the four WC-Co samples were used for determining the grain size of WC and the mean free path in cobalt. SEM micrographs of residual indentations for loads 0.1 - 10 kg on sample WC 15 wt. % Co were used to measure the diagonals of the indents. In figure 3.4 characteristic SEM micrographs of residual indents for three different indenting loads are shown.

3.2.4 Atomic Force Microscopy

A critical analysis of residual microindents requires a surface measuring or profiling techniques with spatial resolution of the order of nanometer. Residual indent measurements involve mapping of the surface topography in and around the indent including the pileups, i.e. material above the polished and unperturbed surface. Residual indents for low indenting loads have diagonals of the order of few micrometers and the height of the pileups along the diagonals is of a few nanometers.

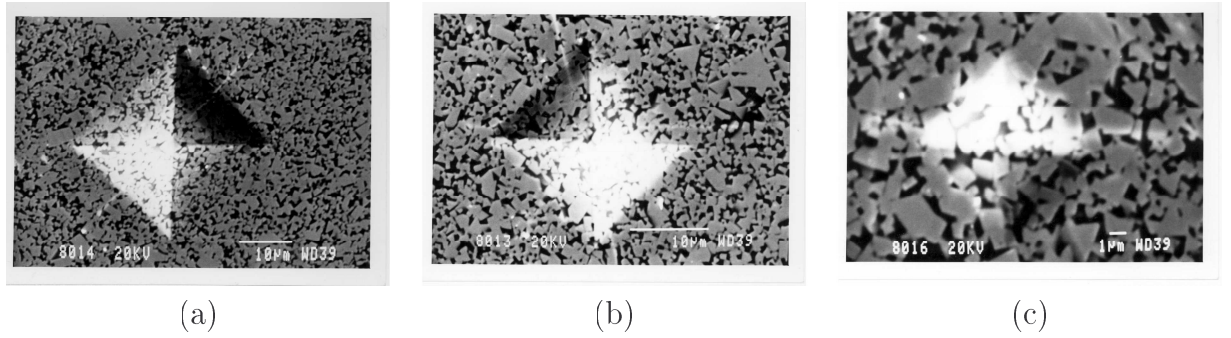


Figure 3.4: SEM micrographs for residual indents on a WC-15wt.%Co sample of (a) 1 kg (b) 500 g (c) 100 g.

In an optical image the dark region defines the residual indent, while the bright-dark fringe demarcate the boundary of the residual indent. The pileups information is absent in both the OM and SEM micrographs. In the SEM micrographs the residual indent is identified by the contrast between residual indent and ‘original’ surface. The pileups in SEM micrographs are not well defined or are not visible as seen in figure 3.4. For the low indenting load (i.e. $<300\text{g}$), the residual indents are not well defined. This can be attributed to the fact that the resolution is poor due to the depth from which secondary electrons are obtained. The surface patterns of topographic origin are often visible purely because of the brightening of edges by ‘leakage’ of secondary electrons from edges as shown by Volbert [Volbert, 1982].

This may be one of the factors contributing to overestimation of the measured diagonal. To resolve these features Atomic Force Microscopy (AFM) with spatial resolution of nanometer scale and lateral resolution of $\text{\AA}$ scale are used. Comparative studies of three dimensional measurement techniques for sub-micrometer (or nanometer) surface perturbations by several authors [Castle and Zhdan, 1997] [Nessler, 1999] [Strausser et al., 1994] [VanHelleputte et al., 1995] , [Lemoine et al., 1999] have show the advantage of AFM measurements compared to SEM. In table 3.2 comparisons of three types of microscopes are given.

AFM is capable of providing quantitative “true” three-dimensional topography of surfaces with spatial and lateral resolution of $\text{\AA}$ scale. AFM is functionally similar to the stylus-based surface profilometry. The main difference is that AFM utilizes much smaller forces between tip and surface, and the size of the stylus or

probe is of a few micrometers in length and with tip radius of a few nanometers. AFM senses forces between the atoms at the tip of the probe and the atoms of the sample. Some of the more commonly measured forces between the atoms at the tip of the probe and atoms of the samples are van der Waals forces (AFM), Born repulsion, electrostatic(EFM) and magnetic forces(MFM), friction and adhesion (LFM, FMM). The magnitude of these forces is of the order of 10^{-13} to 10^{-4} N.

Table 3.2: Comparison of three types of microscopes.

	OM	SEM	AFM
Magnification range	1 - 2000	10 - 100000	200 - 100000000
Resolution x,y [nm]	500 - 1000	1,5 - 3,5	1-10 (tip convolution)
Resolution: z	bad	moderate	0.1nm
Image resolution pixel	2000-10000	2000-3000	256-1024
Environment	air, liquid	vacuum	air, liquid, vacuum
Sample preparation	easy	medium	easy
Conditions	-	conductive surface	-
Noise sensitivity	low	medium	high
Image acquisition time [min]	0,01 - 1	0,1 - 2	1-10

AFM images are a representation of the tip-sample interaction as measured by the deflection of the laser onto the position sensitive photo-detector's (PSPD) in response to the surface topography. In turn, the feedback loop drives the scanner's responses to the applied voltage. The feedback loop is controlled by the electronics and an operating software. Each component of the AFM instrument (scanner, tip, electronics, mechanical mountings, environment, and software) function within their limitations. These factors can be due to either their material composition, mechanical-electrical and physical response to electrical signals and mechanical motion which can influence the observed surface topography.

Any measured image or data that result in an image differing from the actual sample surface is called an artifacts [Lenihan et al., 1995], [Maurice, 1996]. The artifacts can be either due to instruments limitation or physical restriction imposed to the probe-sample interaction. AFM is no exception. Artifacts in AFM can arise - due to mechanical and/or electrical systems, tip-sample interaction, and

computer software (feedback control and image processing algorithms).

The source of artifacts in atomic force microscopy can be broadly categorized into: probe based (1 and 2), physical limitation of instrument based (3,4 and 7), software based (5 and 6);

1. Tip artifacts
2. Artifacts created by samples
3. Scanner and piezoelectric ceramic artifacts
4. Instrument mechanical and electronic design,
5. Electronic controllers and feedback loop
6. Data manipulation
7. Environment and operating condition.

Several books on AFM deal with sources and imaging due to artifacts [Wiesendanger, 1994], [Bonnell, 1993], [Sarid, 1994]. Most manufactures of AFM instruments discuss this aspect in their operation manual.

3.2.5 Acquiring AFM images of Residual Microindents

In this work, the microindents on WC and WC-Co samples were acquired using a commercial atomic force microscope, CP-Autoprobe, Park Scientific Instrument², Sunnyvale, CA, USA. The indents were scanned in Contact-AFM mode with 256×256 pixels scan size, on a $100\mu\text{m}$ scanner with an extensibility of $7\mu\text{m}$ in the Z-axis. Before and after scanning a complete set of indentations (i.e. 1kg - 10g indents) the scanner was calibrated with $9.9\mu\text{m}$ and $0.9\mu\text{m}$ calibration gratings, ensuring stability in the lateral (X- and Y-) direction. The scanning tips (probes) used were Si_3N_4 which are $3.0\mu\text{m}$ long with an opening angle of 70° , fabricated onto a triangular cantilever of length $200\mu\text{m}$, width $22\mu\text{m}$, thickness $0.6\mu\text{m}$ and with a force constant of 0.03 N/m .

²Merged with Veeco Instruments in 2000.

Each of the residual indentations were imaged in 4 scanning directions; (i) along x- axis with the tip probing (a) left to right and (b) right to left; (ii) y-axis with the tip probing (a) top to bottom and (b) bottom to top. Any differences in these four images of the same indentations would essentially be due to the tip geometry, given that all other sources of artifacts are absent.

The images were manipulated with tilt and leveling corrections, with a second order polynomial equalization in the direction of the scan and a zeroth order line by line correction in the direction perpendicular to the scan direction. This correction is essential and is a standard practice for correcting non-linearity and bowing effect in the scanner. The stability of the scanner in the x-y direction was monitored using the calibration grids. The scanners traction and retraction which generates height z-information was calibrated with a 400Å VSNL step-height grid.

Factors such as mechanical vibration, thermal drift and acoustic vibrations that can induce artifacts in the images were eliminated or maintained at minimal value by monitoring the response signal for a 0Å scan size. Where 0Å refers to a point size measurement which would be dependent on the type of scanner.

These images were initially analysed using the program ProScan - Image Processing software, provided by Park Scientific Instrument, Sunnyvale, CA, USA. A detail analysis of the AFM acquired images of residual indents were analysed by the program AutoIndent written specifically for this project, and is described in details in the chapter 4 on Analysis of AFM Images of Residual Indent Imprints.

Chapter 4

Results I: AutoIndent program for the analysis of AFM images of residual indent imprints

4.1 Hardness Value

The computation of the Vickers hardness of a specimen involves calculating the contact area¹ between the indenter and the sample. The size of the contact area depends on the geometry of the indenter and is measured in terms of the diagonal or the depth of the indentation. Usually, the diagonals or the depth are measured after removal of the indenting load, and this can be achieved by numerous surface imaging (or measuring) techniques. In this work the diagonals and the indentation depth are measured by AFM. For one of the samples, WC 15 wt. % Co, the diagonals were measured using optical microscopy (OM), SEM and AFM. The diagonals measured by OM, SEM and AFM shows significant differences, and consequently the hardness values differ by as much as 150% (see figure 5.7). The inherent limitations of lateral resolution in the surface measuring techniques require a critical analysis of the residual indents. Some of the ways in which limitations can be overcome are, by:

1. Measuring the diagonal (and depth) with a higher lateral and vertical reso-

¹This area is referred to by some authors as projected area. In the case of the Vickers hardness H , for an applied indenting load L and area A of the residual indent is $H = L/A$. Where $A = d^2/2 \sin 68^\circ$, d is the measured diagonal of the residual indent, and A is the contact area between the indenter and the specimen. Thus, in this work, this area is referred to as the contact area.

lution technique.

2. Establishing a method of systematically defining the indents, thereafter measuring the diagonal and depth and analysing the surface in and around the indent.
3. Sufficient large statistical samples.

4.1.1 Measuring the Diagonal

Following Meyer's [Meyer, 1908] definition of hardness, hardness is the ratio of the applied load to the projected area of the recovered indent, where "elastic deformation, which is so small as to be susceptible of measurement only by means of refined methods, plays no part in forming the conception of hardness. This definition assumes that there is no elastic recovery of the diagonals² and that one measures the residual indent only, which excludes any contributions from the pileup material around the indent.

In most indentation techniques the indents are measured after removal of the applied indenting load and after recovery of the sample. This raises some fundamental questions:

1. With what accuracy should the diagonals ought to be measured?
2. What constitutes the diagonal of the residual indent?
3. What is the geometric correlation between the indenter and the residual indent?

A critical study of the hardness of a sample should involve a rigorous analysis of the hardness calculated from indents by utilizing techniques with as high precision and spatial resolution as possible. Despite improvements in lateral resolutions with measuring techniques (e.g. SEM, AFM, Micro-Raman,...), the diagonals (or diameters) are still largely measured in industry by optical microscopy for practical convenience.

²For pyramidal indenters the diagonal is measured, while for cone or spherical indenters the diameter is measured.

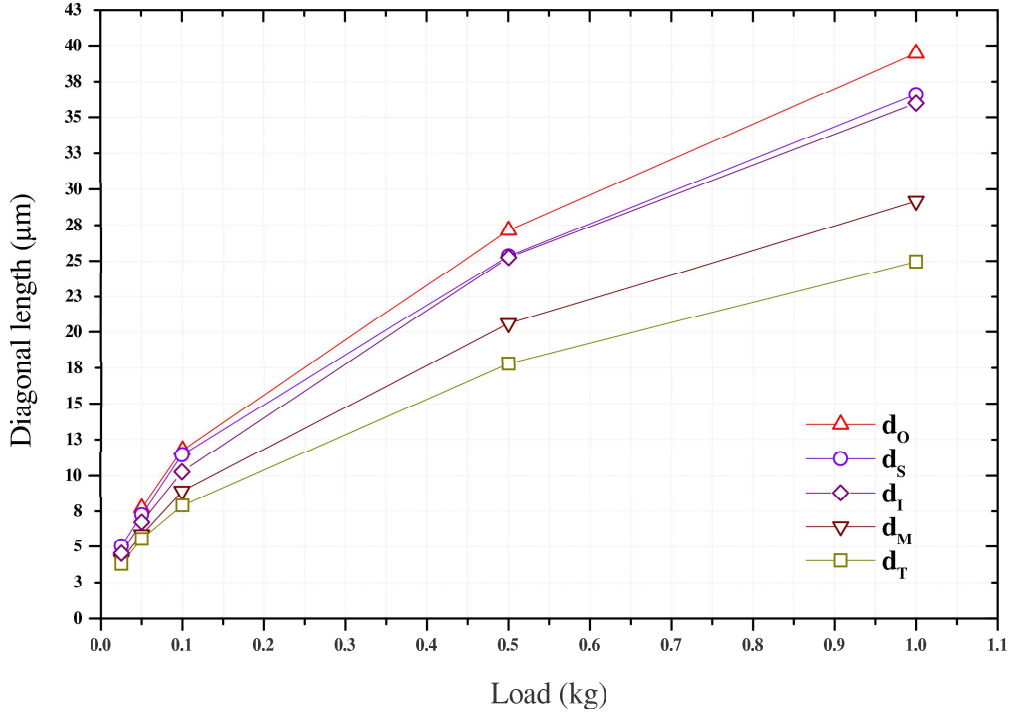


Figure 4.1: Length of the diagonal of the residual indents in WC-15 wt.% Co for applied indenting loads 25 gm - 1 kg measured by OM (d_O), SEM (d_S) and AFM (d_T , d_M and d_I).

Preliminary investigations of recovered micro-indents on WC 15wt.% Co of loads 50 g-1 kg were done using optical microscopy (OM), differential interference contrast microscopy (DIC), scanning electron microscopy, and atomic force microscopy (AFM). In table 3.2 a comparison of the resolution capabilities of each of the techniques is given.

Figure 4.1 shows the values of the diagonals as measured by the three techniques. Evidently, the value of the diagonals differ by up to 33% between OM and AFM, and by up to 5% between OM and SEM. This leads to significant differences in the hardness.

To understand these discrepancies, three-dimensional topography AFM images were rigorously scrutinized with emphasis on the region around the corners of the residual indents and the points between which the diagonals are measured we re indicated in figure 4.2. Extending the line profile along the diagonal shows the

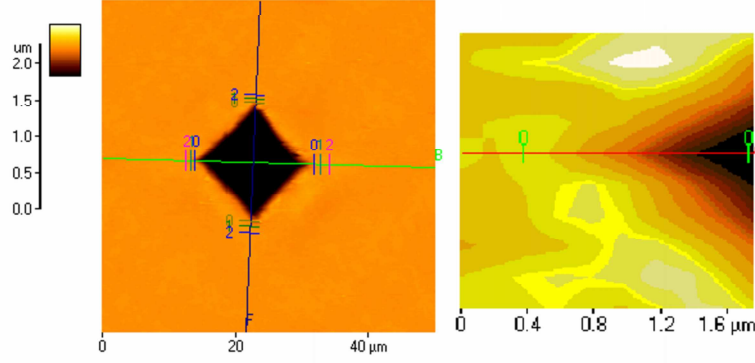


Figure 4.2: An AFM image of residual indentation. The line profile is obtained by selecting data points near the opposite corners of the indent. The diagonals are then measured by selecting pair of points on the line profile (for example the length of the diagonal could be defined by any one set of pair of points 0, 1, 2) on either side of the corners. The image on the right is a magnification of the region around one of the corners from the image on the left.

pileups. Two pairs of points are distinct - firstly, the maximum vertical point of the pileup with respect to the apex of the inverted pyramidal shaped indenter, and secondly a point exists in the pileup – “original” surface interface, this point is frequently referred to as including pileups (I) in this text. Figure 4.1 also shows the length of the diagonals measured between the maximum vertical points (M) referred to as d_M , and d_I is the length of the diagonals measured between the points I.

Comparing the diagonals d_M and d_I with the diagonals d_O an average difference of 25% and 11% respectively is observed. The difference between the diagonals d_I , d_S and d_O can be attributed to the lateral resolution of the three techniques. For the AFM analysed indentation images three distinct pairs of points are identifiable, namely d_T , d_M and d_I .

4.2 ProScan Analysis

AFM images were acquired in 256×256 pixel scan size mode. The data for each image are truly 3-dimensional, containing height information for each x-y location scanned and are displayed in gray scale format. In a gray scale image, the brightness corresponds to vertical height on the surface. The gray scale display of data is also a 3-dimensional rendition of data. The higher points are brighter, and lower points are darker.

The image processing software, ProScan Image Processing Ver. 1.5 [PSI, 1995] was provided by the manufacturers. The features of ProScan are (i) image enhancements (ii) measurements of topography features and (iii) presentation of images in 2D and 3D.

1. The image enhancement technique includes

- a) Flattening - An image may have a curvature or slope which may be due to an artifact induced by the motion of the scanner during acquisition. This curvature or slope of the image can be corrected for by applying an n^{th} order polynomial fit either to the entire image or as line by line (considering the image to consists of data points as 256 column by 256 rows i.e. a matrix of 256 x 256 data point). In the line by line method the coefficients of the polynomials changes with every line being corrected for, while in the entire image method the coefficient of the polynomial are as an average of the entire image.
- b) Filtering - the primary function of this routine is to remove artifacts (or noise) from the image with regular periodicity or features. Fast Fourier Transforms (FFT) is applied to the original image. The relevant frequency of the noise is eliminated by either applying Wiener filter or x- and y- band pass or radial band pass filters. The new FFT matrix formed is without the noise frequency. Thereafter inverse-FFT is applied to new FFT matrix which generate an enhanced image with minimal artifacts component in it.

2. Analyze - features in the image are analyzed individually or as an entire image. The two main forms of measurements are available (i) line mea-

surements (ii) region measurement. This will be illustrated in detail in the following section 4.2.1 and 4.2.2

3. Display - allows for several images to be displayed simultaneously or display the image as a 3-dimension image.

4.2.1 Line Measurements

AFM images consist of 3D topographs which are analyzed by surface or line measurements. The selection of features or points of interest are selected from the 2D AFM image only. The display contrast of the image can be enhanced by varying the gray scale threshold position [Note: This only changes the display image and not the actual data.]

In this section only line measurements are discussed. A line profile is obtained between two selected points on the image displayed in 2-D. The line profile constructed between these points is actually a height profile. For a given line profile, the parameters that can be measured between a pair of points (or markers) are:

- (i) Distance and spacing.
- (ii) Height or depth.
- (iii) Gradient of the line formed between the selected pair of points (markers).
- (iv) Average roughness along the line.

The distinction between spacing and distance is, that the spacing is the horizontal separation between the two points (markers), while distance takes into account the height difference as well as the horizontal separation between the points. The average roughness along the height profile is given by $R_{ave} = \sum_{n=1}^N \frac{|Z_n - \bar{Z}|}{N}$, where $\bar{Z}$ is the mean height, Z_n is the height of the n^{th} data point and N is the total number of selected data points in the line.

Figure 4.3 displays a typical line profile across the indent with the measurements analysis between a pair of markers (or points). Some of relevant measurements are marked in circles. They are (a) the distance between the points, (b) angle along

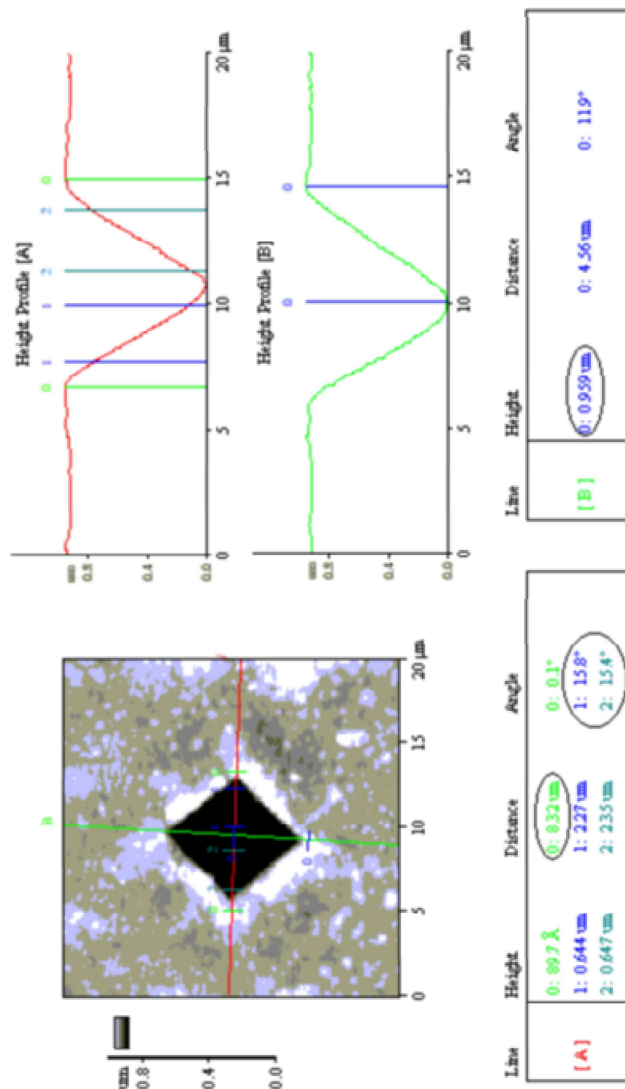


Figure 4.3: A typical line profiles across the indent as measured by the software ProScan. The three parameters of interest for the indentations are height (depth), distance and angle. The ringed numbers illustrates the parameter measured depends on the positioning of the markers. The numbers ringed in the table labeled Line [A] gives the distance and angle between points marked 0 & 0, 1 & 1 and 2 & 2. While in line [B] the height difference between the two markers is ringed, in the given case, the ringed value is the depth of the residual indent.

the indent profile (c) maximum depth of the indent (line B) is obtained by placing one of the marker at the minimum Z-point.

4.2.2 Surface Measurements

The second set of analyses consists of surface measurements. In this routine the region of interest (ROI) is selected by demarcating the boundaries around the feature of interest which can either be included or excluded in the measurements. Within the selected region the parameters that can be measured are:

(i) Roughness

- (a) Average roughness $z_{ave} = \sum_{n=1}^N \frac{|z_n - \bar{z}|}{N}$, where N is the total number of points in the selected region.
- (b) Root-mean-square roughness - is given by the standard deviation of the data. $z_{rms} = \sqrt{\sum_{n=1}^N \frac{(z_n - \bar{z})^2}{N-1}}$.

(ii) Height

- (a) Maximum peak-to-valley height separation within the selected region, $z_{p-v} = z_{max} - z_{min}$.
- (b) Mean height, given by the average height of the selected areas.
- (c) Median height, the height value which divides the height histogram into two equal areas.

- (iii) Surface area measurements - The surface area is approximated by doubling the area of the triangle connecting the data point(pixels) to two of its nearest neighbors taking into account the height difference between the points. The resulting rectangular areas corresponding to each of data points in the selected region are then summed up, which gives the surface area of ROI. The surface area determined by doubling the triangle area is an approximation which assumes symmetrical distribution of the nearest neighbourhood points.

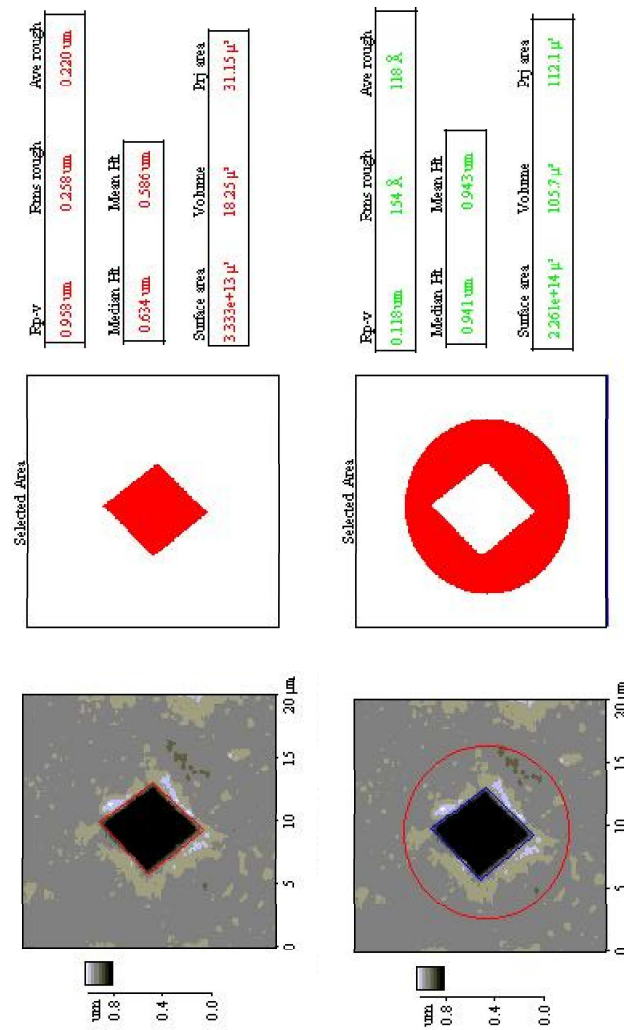


Figure 4.4: Surface analysis as measured by ProScan. The figures at the bottom are the images as acquired, the images in the middle show the selected region, while the tables on top to the images give the various parameters determined by ProScan.

- (iv) Volume measurements - gives the volume beneath the ROI. The volume measurement is given by summing the difference in height of each data points with respect to the minimum height (zero reference) within the entire the image. The zero reference is the minimum data point of the entire image and not just of the ROI.

Figure 4.4 gives the surface analysis relevant to this work. The figures on the left relate to the measure of the residual indent, while the figures on the right relate to the region of the pileup. The figure on the left is used to demarcate the selected region within the image, while the figure in the middle shows the selected region(shaded). The relevant parameters are volume of pileups and residual indent, surface area, R_{p-v} -height value (equivalent to the depth measurements from line profile) and average roughness within the ROI.

4.2.3 Measuring using ProScan and its Limitation

The measured parameters obtained using the ProScan program are useful as preliminary results for the analysis of residual indentations. Although the image corrections (flattening and filter) of the raw data are achieved quite effectively by ProScan, the line and surface analyses are not satisfactory. The deficiencies are due to the nature of the features of interest in this particular project, rather than the program itself. There are a number of factors that influence the ‘true’ results, some of these are:

- (i) Corner points of the indent.
- (ii) Points between which the diagonal is to be measured.
- (iii) Points used to measure the depth of the indent.
- (iv) The angle measured along the diagonal-line profile does not necessary pass through the apex of the indent.
- (v) Limitations in defining the residual indent and the extent of the pileups.

4.2.3.1 Corner Point

The corner points of the indent are determined by magnifying the region around the indent's corners. The corner points identifying the indent

The magnified image may still consist of 100's of pixels points from which to select a point that correctly identifies the corner point. This process is repeated for the opposite corner of the indent. Once the opposite corner points have been selected the line profile is then drawn through them.

4.2.3.2 Diagonal measurements

Once the line profile has been obtained, the next step is to determine the pair of points between which the diagonal is measured. As a practical guide see figure 4.5, these points are determined with the aid of two markers, by placing one of the marker (0) far off the opposite corner, representing the 'original' surface. The 1st marker is then moved around the corner of the indent-surface interface region such that the height difference between the two markers is minimum. This position of the marker is then selected as the first point. The second point is similarly determined by placing one marker (1) at the first selected point while moving the second marker around the opposite corner of the indent-surface interface region, again until a minimum height difference is observed. This markers is the second point. The distance between these two points (markers 2) gives the length of diagonal of indent.

4.2.3.3 Depth of indent

Once the length of the diagonal has been determined, the diagonal points are then used to determine the depth of the indent (see figure 4.3 height profile B). By keeping one marker of the diagonal point fixed and moving the second marker along the diagonal above the lower section of the line profile, the depth is the maximum height difference value is observed. The same process is repeated with the other set of diagonal points. The mean of four such depth values are used to determine the depth of the residual indent.

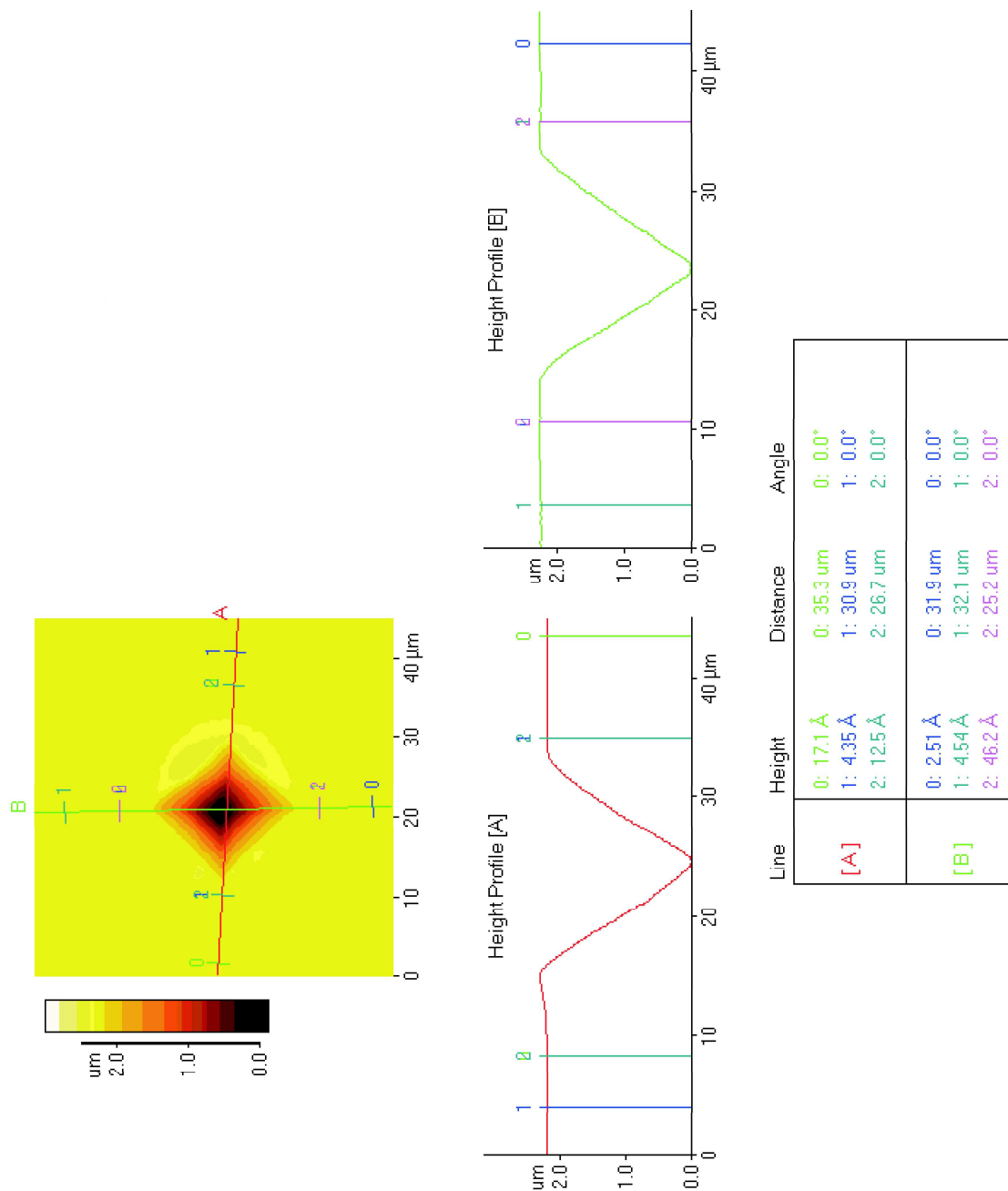


Figure 4.5: Points selected between which the diagonals are measured. Pair of markers are 00, 11 and 22. The length of the diagonal is between 22.

4.2.3.4 Angle measurements

The angle along the edges of the indent are determined by placing randomly three pairs of markers along each slopes of the ('V') section of the diagonal line profile, in figure 4.3, height profile (A) shows markers 11 and 22, and the angles measured between the markers 11 and 22 are ringed. Measuring of the angle along the 'V' of the diagonal line profile encounters two problems: (i) firstly - the segment of the line profile along the edges of the indents are curves, thus the angle measured is a secant. (ii) secondly, the diagonal line profile obtained from the selection of the corner points does not necessarily pass through the apex of the indent, nor does it follow the indent edges.

4.2.3.5 Limitations

The limitations of ProScan are not restricted to line measurements but also exists for surface measurements. Selection of the region that defines the residual indent and the pileups is very subjective and randomly repetitive. Thus the volume and the surface area of the residual indent and pileups calculated varies. The surface area is calculated by doubling the area of the triangle formed about a point connecting it to two of its nearest neighbouring points and summing it for all the points within the selected region. The volume is calculated as the product of the projected area and the mean height of the selected region.

The calculated volume of the residual indent is the volume of a cuboid with a pyramidal cavity in it and is not the volume of the residual indent as required. While, the volume of pileups is over estimated as the mean height used is the actual height and not the mean height within the selected region.

Limitations of ProScan are largely due to the subjectivity involved in determining the relevant points. In addition the diagonal line profile does not pass through the apex of the indent. Thus the angle measured does not necessarily represent the half-apex angle of the indent. Both the surface area and the volume are erroneously calculated. The surface area is estimated based on the selection region defining the indent and the extent to which the pileups are formed.

4.3 Program - AutoIndent Analysis

Despite developing a "systematic" routine in analysing the data using ProScan, the level of subjectivity involved is relatively high, thereby increasing the error with which the data are handled. To overcome these and other limitations of ProScan a program was written in MATLAB V.4.1 to analyse the data with a mathematical basis for selecting the points and their analysis. In this section the principle underlining the analysis of program is given. The entire program is given in appendix D, and a recently published paper [Shrivastava and Luyckx, 2007] on the typical results that can be obtained from the program is given in appendix H. The program is be divided into three sections:

- (i) Obtaining the line profile.
- (ii) Identifying points between which the diagonal is to be determined.
- (iii) volume measurements of the residual indent and the pileups surrounding the indent.

Obtaining the line profile involves reading the PSI data file, thereafter identifying the edges of the indent. PSI data files are stored in hierarchical data format (HDF). HDF is a data file format designed by the National Center for Supercomputing Applications (NCSA) at the University of Illinois at Urbana-Champaign. HDF is a platform independent file format, which supports a variety of data types: scientific data arrays, tables, and text annotations, as well as several types of raster images and their associated color palettes. This makes it possible for programs to obtain information about the data from the data file itself, rather than from another source.

HDF files are also self-describing. HDF standardizes the format and descriptions of many types of commonly used data sets, such as raster images and scientific data. For each data object in an HDF file, there are predefined tags that identify information such as the type of data, the amount of data, its dimensions, and its location in the file.

The self-describing capability of HDF files has important implications for processing scientific data. It makes it possible to fully understand the structure and

contents of a file just from the information stored in the file itself. A program that has been written to interpret certain tag types can scan a file containing those tag types and process the corresponding data. Self-description also means that many types of data can be bundled in an HDF file. For example, it is possible to accommodate symbolic, numerical, and graphical data in one HDF file.

During the time the AutoIndent program was being developed there were no routines available in the public domain or commercially that could import scientific data (AFM image) and its parameters directly into Matlab. A sub-routine was written to read the PSI-HDF files, extracting information about the scan size, z-calibration factor and image-data, which are essential for the analysis of the indentation image-data. Once the image-data had been read by Matlab the next step of the analysis begins.

4.3.1 Mapping the Line profile

Mapping out of the line profile is achieved in three stages. Firstly, the apex of the indent is obtained by determining the minimum z-value point in the image. This point is referred to as the apex. Once the apex has been determined, thereafter using the Sobel operator edge detectors method [Sobel, 1970] the corner points defining the edges of the indent are determined. At a given depth of the indent, the edge detection gives a “square”, the corner points of which are used to define line profile along the edge of the indent. The four sub-line profiles are constructed between the apex point through the corner points to the edge of the image (see figure 4.6. Each of these sub-lines are extended in their respective quadrants. The opposite sub-line profile are combined to generate a line profile along the edge of the "inverted-shape pyramid" indent forming the line profile acting along the diagonal. Each of these line profile consists of 256 points.

4.3.2 Point Locators

Once the line profile has been obtained, the next task is to determine points that are characteristic of the indent. There are four distinct points that play a key role in defining the indent.

- (i) Apex point of the indentation - This point represents the reflection of the tip

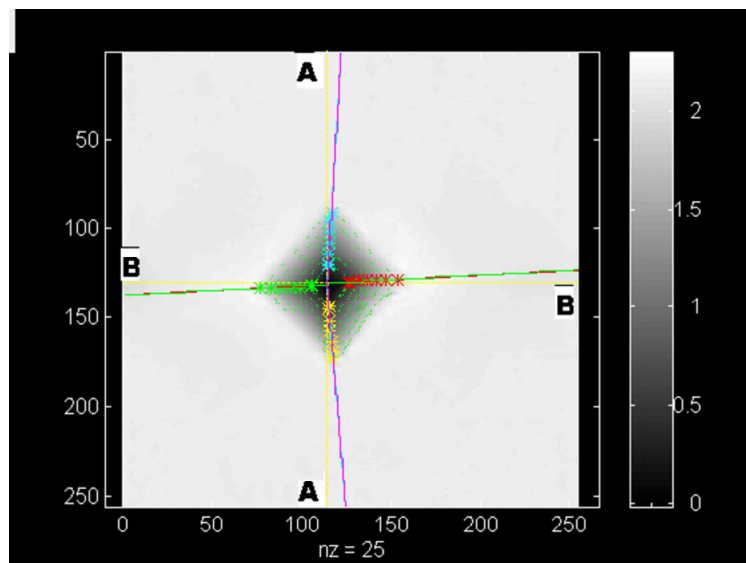


Figure 4.6: The line profile is obtained by combining sub-line profiles between the apex point and lines along each of the edges of the residual indent. The straight line marked AA and BB are obtained by ProScan. All the other lines are using the AutoIndent program.

of the indenter. Even after recovery it is expected this point is correlated to the tip. Although the geometry of the Vickers indenter does not necessarily generate a single point (apex). Geometrically four planes do not necessarily meet at a point, but as a line. SEM micrographs figure 3.2 show this to be true, but the tip of the indenter is effectively considered to have a radius of curvature, there will still be a minimum point that can be used to represent the apex of the indent. In any given image there is only one point (x_0, y_0) which has the lowest z-value, in cases, where there are more than one point with the same lowest z-value centre of these points is considered to be the apex of the indent. The apex point is simply determined by finding the lowest z-value in the image.

- (ii) Gradient point or 'Turning point' - A closer look at the recovered indent line profile shows a point of inflection which undergoes a gradient change. This is a very distinct point where the recovered surface almost coincides with the height of the 'original' surface. Although this is not necessarily the

case, more so when sinkin occur. This point would also mark the onset of pileups formation. For references, this point is referred to as the turning point abbreviated using T. This point (x_T, y_T) is determined by the gradient method as the name suggests.

- (iii) Maximum point - Zooming closer around the residual indent-‘original’ surface region shows a pileups like feature, which is a characteristic of the sample. Invariably, there is a point (x_M, y_M) on this pileups with a maximum height (z-value is maximum). This point is referred to as the maximum point, abbreviated as Max.
- (iv) Including pileups - Examining the pileups, a tailing effect is observed of the pileups on the side that merges into the surface unperturbed by the indentation process. This point (x_I, y_I) defines the extent of the pileups, and is determined by the gradient method. The point is referred to as including pileups, abbreviated as I. In principle, the z-value of this point should equal to the z-value of the unperturbed surface, this might not always be the case. As the tailing point and the surface are at the same height. At angstrom (atomic) scale the surfaces are never really flat. Thus, an average z-values of the unperturbed surface is determined by excluding region of the residual indent, pileups and up to 20% of the region around the pileups³. To cross-check the z-value of the I obtained with the gradient method. In cases, where this section of the line profile had as high roughness, a median filtered differences method [Matas et al., 1995] is be used.

Once the (x,y,z) co-ordinates for each of these points for the sub-line profiles are known a host of information can thereafter be determined (i) length of the diagonal, (ii) depth (height), (iii) angle along the opposite edges of the indent and (iv) length of pileups.

Three lengths of diagonals are determined d_T , d_M and d_I , and their corresponding depths h_T , h_M and h_I . In addition to the diagonal and depth, the half-angles along different section of line profile can also be determined eg. α_{O-T} , α_{T-M} and

³20% is chosen randomly, after qualitative analysis of several images the extent of the pileups contribution along the diagonal was found to be less than 10-15%, thus a 20% extent was assumed to be the extend of the pileups.

α_{M-I} . Using the length of the diagonals, the total length of the pileups can now be determined.

4.3.3 Volume measurements

The two volumes of interests are:

- (i) volume of the residual indent (cavity in the sample).
- (ii) volume of the pileups.

4.3.3.1 Volume of Residual Indent

The boundary of the residual indent is defined by the points T. The average height of a plane drawn between the four T points is taken to be the reference height Z_{RI} . The volume of the residual indent used in the AutoIndent program is given by

$$V_{RI} = A_P \left(\sum_{i=1}^{N_{RI}} (Z_{RI} - z_i) \right)$$

where N_{RI} is the number of pixel points within the residual indent, $A_P = \text{scan size} / 256$ is the area of each pixel, $A_{proj.}$ is the projected area and z_i is the height of the i^{th} pixel point. Unlike ProScan measurement this does actually give the value of the residual indent.

4.3.3.2 Volume of Pileups

The volume of the pileups - The pileups are defined as the region between the points T and I. Since the extent to which the surface is perturbed during the indentation process is not well established, the region considered for calculating the pileups volume is assumed to be more than that defines by the points I. The extent of pileups region is considered to be annular figure 4.7, with a radius of 20% more than half the average distance between the opposite points I i.e. $r_{PIJ} = 1.2d_I/2$. The factor of 20% is chosen with the assumption that at these points the surface perturbation after indentation are negligible. The volume calculated using r_{PIJ} includes contributions due to surface roughness, and any surface elastic recovery effect that might occur during the indentation process.

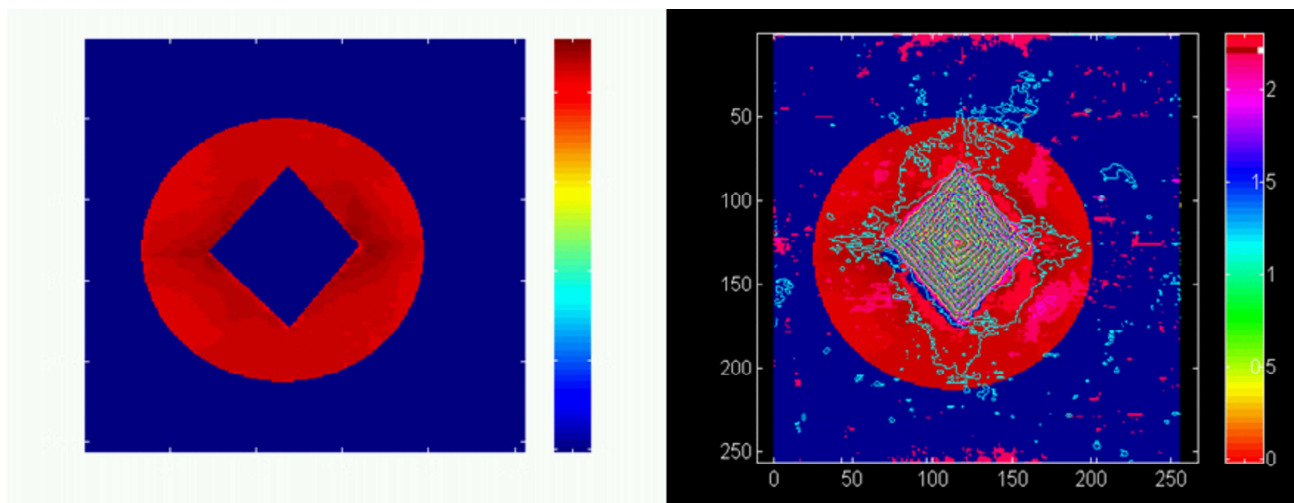


Figure 4.7: Region for which the volume of pile-ups and volume residual indents are determined. The square in the centre represents the residual indent and the circle represents the extent to which the pile-ups are determined. Image on the left is an overlap of the topography onto the region of pile-ups and residual indent selected.

The volume of the pileups corrected for the surface roughness is now given by

$$V_{PU} = A_P \left(\sum_{i=1}^{N_{PU}} (z_i - Z_{bkg}) - N_{PU} z_{rms} \right)$$

where N_{PU} is the number of pixel points within the pileups region, and Z_{bkg} is the vertical height of the surface at the point where the surface is unperturbed by indentation.

Chapter 5

Results II: Results of measurements

In this chapter the results of measurements from a total of 144 indentations, with each indent acquired in 4 AFM scanning directions are presented. The results related to the elastic recovery of the indent when the indenter is unloaded are presented in appendix E.

5.1 The Indentation Line Profile

5.1.1 Line profile

A typical line profile along the edge of the residual indent obtained by the AutoIndent program is shown in figure 5.1. The line profile shown is the ‘true’ trace profile along opposite edges of the residual indent passing through the apex of the inverted ‘pyramid’. The apex, point ‘O’ in figure 5.1 is identified as the point of minimum vertical height within the given AFM image and in the line profile. In the line profile (see figure 5.1), three pairs of distinct points marked "T and T'", "M and M'" and "I and I'" are defined. The two pairs of points T & T' and I & I' were determined by using the gradient method described in chapter 4 while the third pair of points M and M' are the maximum height in the line profile on either sides of point ‘O’. The primed and the unprimed notation are used for points on opposite sides of ‘O’. OT, TM, MI, OT', T'M', are M'I' are curves with different radius of curvature (although due to different scales on the x- and y- axes in the line profile OT and OT' appear to be straight lines).

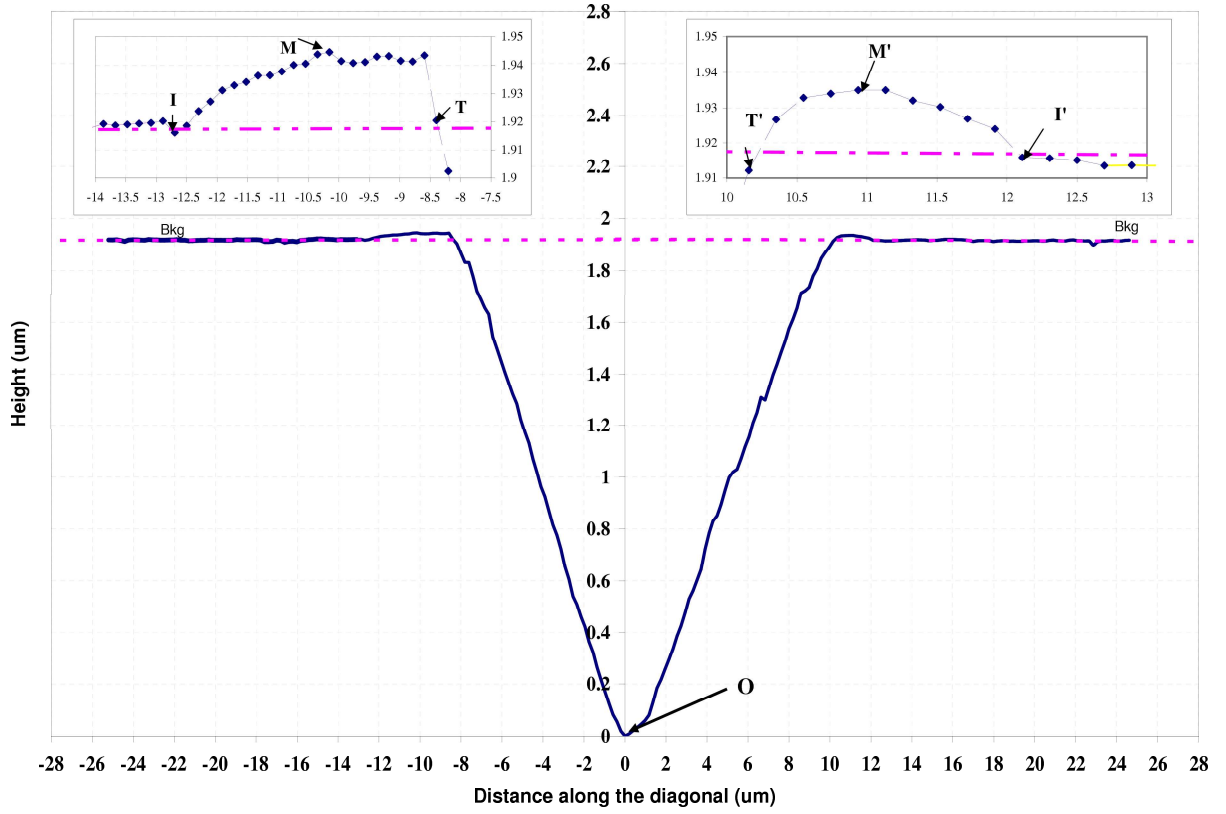


Figure 5.1: Line profile along the opposite edges of a residual indent obtained by AutoIndent. The y-axis gives height with respect to the minimum point 'O'. The 'V'-part of the line profile is along the opposite edges of the residual indent, and the dotted horizontal line represents the 'original' surface. Points T, T', M, M', I and I' are defined in the text. The inserts shown are the section of the line profile representing the magnified pileups section on opposite sides of the indent.

5.1.2 Comparison of the diagonals measured by OM, SEM and AFM

For the sample WC-15 wt% Co, the diagonals of all the residual indents after unloading were measured by OM. The diagonals for the same residual indents were measured from SEM micrographs of the residual indents and compared with those measured by OM. In addition to OM and SEM measurements of the diagonal, the same residual indents were measured by AFM in contact mode. The diagonal

lengths were calculated between the pair of points I and I' (d_I), M and M' (d_M) and T and T' (d_T) (see figure 5.1 page 50) using the program AutoIndent (section 4.3). Figure 5.2 shows all the diagonals measurements for the WC 15 wt% Co samples for applied indenting loads 25g to 1kg.

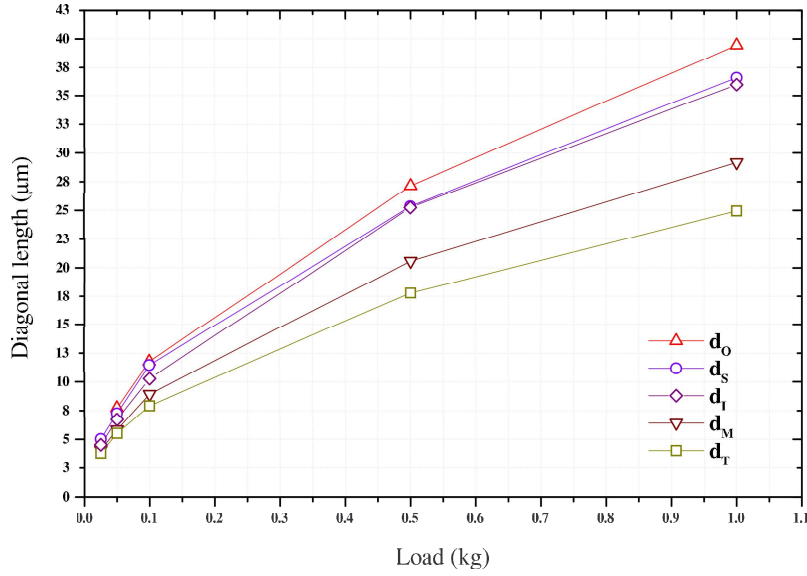


Figure 5.2: Length of the diagonal of the residual indents in WC-15 wt.% Co for applied indenting loads 25 gm to 1 kg measured by OM (d_o), SEM (d_s) and AFM (d_T , d_M and d_I).

5.2 Results from AFM images analysed using the AutoIndent program

The results from the AutoIndent program are divided into line profile, volume and pile-up measurements. In this section, results from the line profile measurements of the diagonal, depth and angle are given.

5.2.1 Diagonal

The diagonals measured using the program AutoIndent (Chapter 4) for indenting loads 50g - 1 kg for the four grades WC-6 wt.% Co, WC-11 wt.% Co, WC-15 wt.% Co and WC-20 wt.% Co are given in the figure 5.3. In figure 5.3, three

different diagonals for each indent, identified by the pair of points 'I & I' (d_I), 'M & M' (d_M) and 'T & T' (d_T), are given. The hardness was evaluated by using these sets of results. The diagonals d_T , d_M and d_I are further used in section 5.5 to characterize the shape and extent of the pileups.

5.2.2 Depth

The indentation depth of the residual indent is the vertical distance between the 'original' unperturbed surface and point 'O' (see figure 5.1 page 50) defined as the height difference between points I and 'O' (see figure 5.4), where point I (and I') are at the level of the 'original' unperturbed surface. The height differences between points M (and M'), T (and T'), I (and I') and 'O' can also be seen in figure 5.1 because they are later used for quantitative characterisation of pileups.

5.2.3 Angle

The AFM images allow for 3-D topographical analysis of the residual indents, and the measurement of the apex angle $\widehat{TOT'}$. The angle $\widehat{TOT'}$ is the angle subtended by the secants between OT and OT'. The results given in figure 5.5 (page 54) are for the half-apex angle between the opposite edges of the residual indent for all the grades of carbides at various indenting load. Despite a large scatter in the results a significant deviation from 74.05° (half-angle of the Vickers' indenter along the diagonal) is observed.

In figure 5.6 (page 55) the angle between the plane parallel to the 'original' surface containing the apex point 'O' and secant between point 'O' and each of the data points in the AFM image are shown. The figures were generated by the AutoIndent program designed for this project. For the Vicker's indenter, there are two angles of interest: (i) 136° - angle between opposite faces, (ii) 148.1° angle between opposite edges. The faces of the Vicker's indenters are plane [Hasche et al., 1998] (see figure 3.2 page 23).

Each data point of AFM images consists of three dimensional information, with x and y giving the position of the point in the image and z the height, which can be translated into spherical coordinates with point 'O' of the residual indents' AFM images as the origin for the spherical coordinates (r, θ, ϕ as radial, angle and

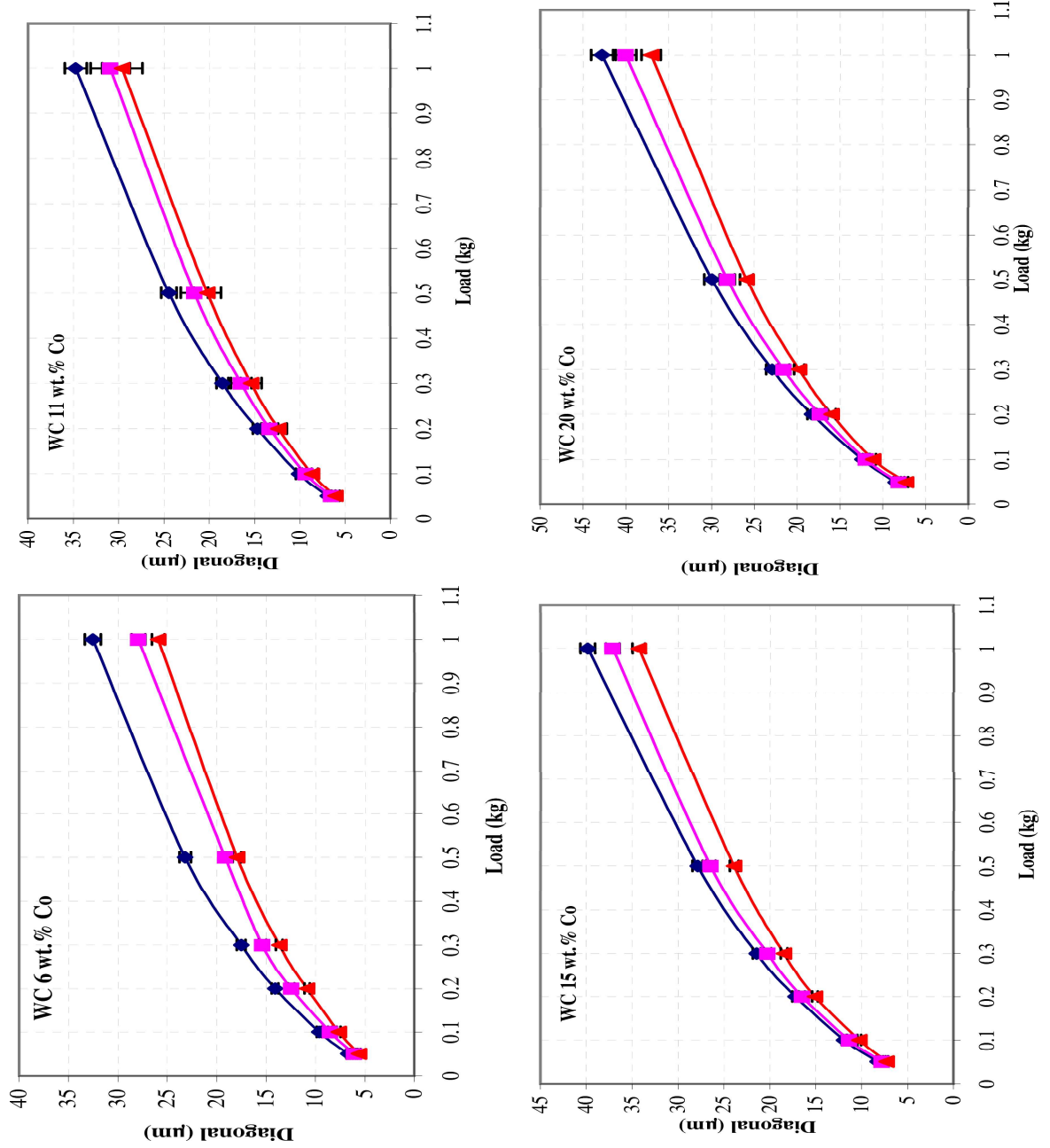


Figure 5.3: “Diagonals” d_T ($\blacktriangle$), d_M ($\blacksquare$) and d_I ($\blacklozenge$) measured by AutoIndent for all the four cemented carbide grades are given.

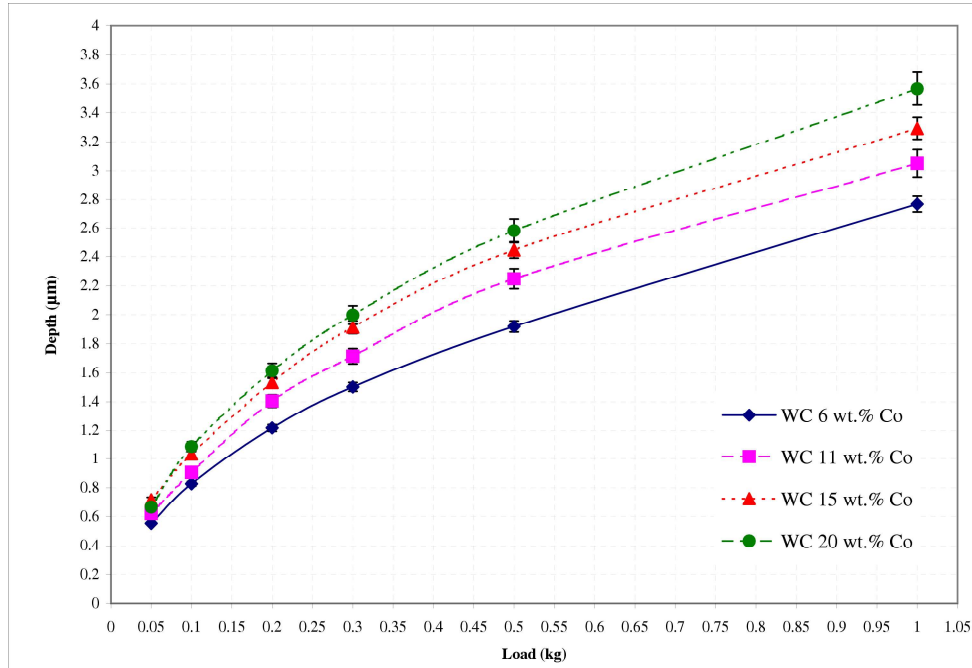


Figure 5.4: Depth of residual microindentations as measured by AFM for the WC-Co grades tested.

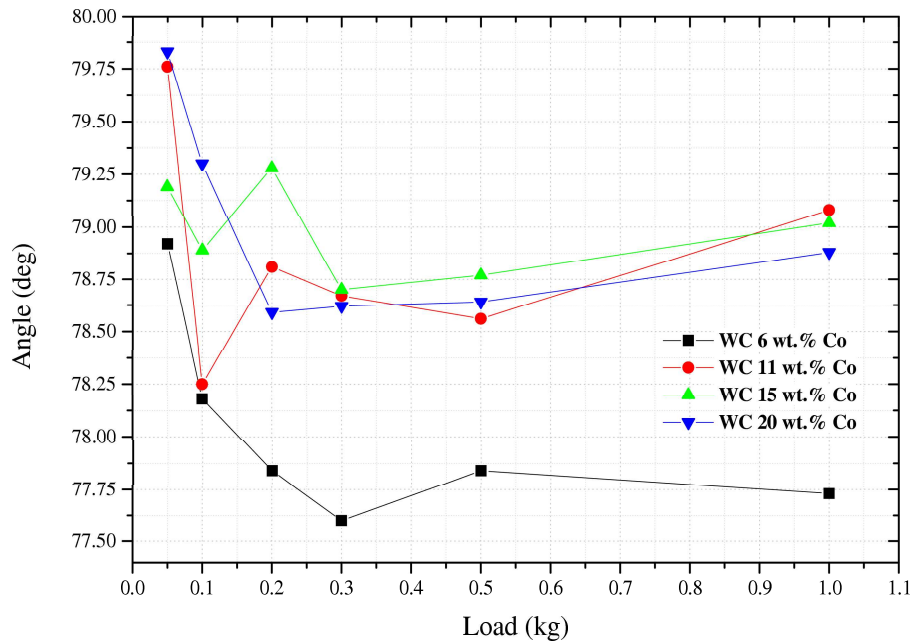


Figure 5.5: Average half-apex angle measured along the opposite edges of the residual indents in the WC-Co grades tested.

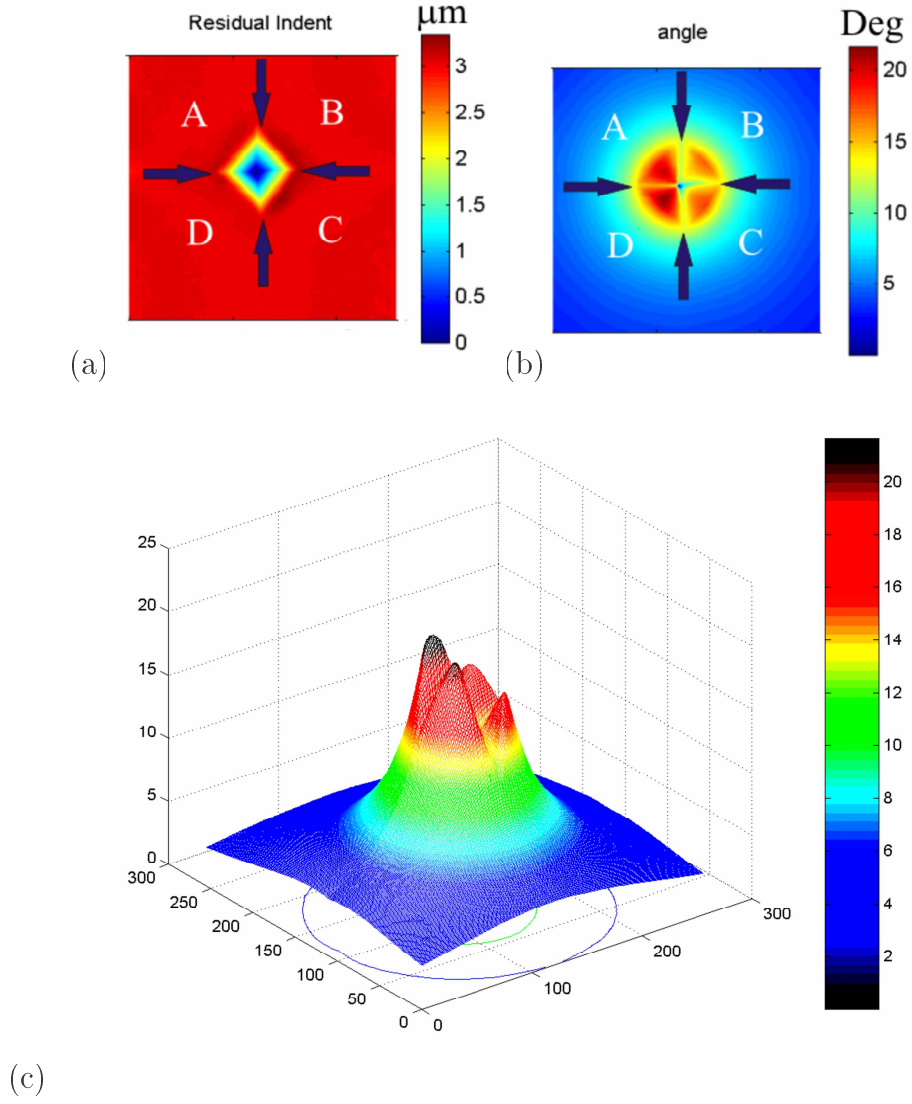


Figure 5.6: (a) is the AFM image of residual indent, while the azimuthal angle image (b) is with respect to minimum height point (point 'O' of the residual indent). The solid arrows indicate the direction along the edges of the indent and letters A,B,C and D refer to the faces of the indent. (c) Azimuthal angle image in 3-D, the points of maximum angle along each faces are the peak point or maximum angle, as explained in details in the text.

azimuthal angle) system. Of relevance for this work is the azimuthal angle.

In figure 5.6 (page 55), figure 5.6(a) is the AFM image of the residual indent and figure 5.6(b) is the azimuthal angle image for the same indent. Comparing images (a) and (b): the AFM image shows the size of the residual indent and its surroundings without any substantial information about the shape of the indent faces and edges. While the azimuthal angle image has four distinct features: (i) the dark dot in the centre is the apex point of the residual indent AFM image (ii) from the centre, four straight lines correspond to the edges of the residual indent (iii) between the edges is the region of varying contrast which represents the faces of the residual indent, (iv) the region of concentric rings correspond to the region surrounding the residual indent.

In the given 2-dimensional azimuthal angle image, the edges are easily identified and are indicated by the solid arrows. While, the region between the edges for each of the quadrants A, B, C and D, shows a non-uniform contrast, a feature not easily visible in the residual indent AFM image (a). A three dimensional representation of the azimuthal angle image (figure 5.6(c)) shows four peaks for each of the faces of the residual indent. The peaks also show that there is non-uniform recovery of the material and the point with the highest z-value is the point of maximum azimuthal angle. The tips of the peaks are the points of maximum curvature of the face. The residual indent image of the inner faces and edges is found by AFM to be of convex shape. The ridges between the peaks are along the edges of the residual indent. The variation in the peaks height can be attributed to non-homogenous material.

5.3 Variations of Indentation Hardness

The results of the hardness evaluated from the measured diagonals and depths for the samples are given. These results show the indentation size effect in cemented carbides (WC-Co) for varying cobalt content and approximately constant WC carbide grain size.

Table 5.1: Values of diagonal and depth measured by AutoIndent and the evaluated hardness.

Load (kg)		1	0.5	0.3	0.2	0.1	0.05
WC 6wt.% Co	Diagonal (μm)	32.5	23.2	17.5	14.1	9.6	6.7
	Depth (μm)	2.8	1.9	1.5	1.2	0.8	0.6
	Hardness (GPa)	17.7	17.9	18.3	18.8	20.1	20.8
WC 11wt.% Co	Diagonal(μm)	35.2	24.0	18.8	15.3	10.7	7.3
	Depth (μm)	2.9	2.2	1.7	1.4	0.9	0.6
	Hardness (GPa)	14.7	15.3	16.4	17.1	17.8	18.5
WC 15wt.% Co	Diagonal (μm)	39.9	27.9	21.4	17.2	11.9	8.3
	Depth (μm)	3.3	2.5	1.9	1.6	1.0	0.7
	Hardness (GPa)	11.7	12.0	12.1	12.6	13.2	13.6
WC 20wt.% Co	Diagonal (μm)	42.6	29.9	22.9	18.3	12.6	8.9
	Depth (μm)	3.6	2.6	2.0	1.6	1.1	0.7
	Hardness (GPa)	10.2	10.4	10.7	11.2	11.8	12.5

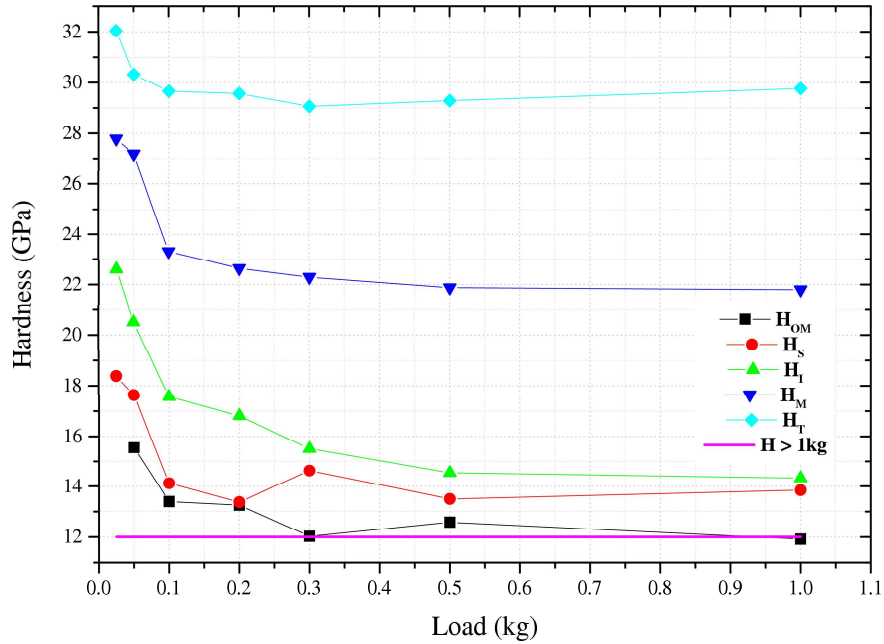


Figure 5.7: Influence of variation in diagonals measured by different techniques on the evaluated Vickers hardness for WC 15 wt% Co, where H_{OM} and H_S are the hardness evaluated using the diagonals measured by OM and SEM respectively. The hardnesses H_I , H_M and H_T are evaluated using the diagonals d_I , d_M and d_T respectively, and H ($\geq 1\text{kg}$) is the macrohardness of the sample as measured by OM.

5.3.1 Hardness dependence on the measuring technique of the residual indent

It is crucial in validating the hardness measured, to assess the ‘true’ diagonal defining the indent. From the results of the diagonal lengths measured (figure 5.2) (page 51) by optical microscope, scanning electron microscope and atomic force microscope (OM, SEM and AFM) for the WC-15 wt% Co sample, the hardness evaluated is given in figure 5.7 (page 57). The bold solid line in the figure 5.7 represents the measured hardness of the sample for indenting loads $\geq 1\text{kg}$, that is the macrohardness, assuming that the macrohardness is a constant and independent of indenting load.

5.3.2 Hardness variation when calculated in terms of depth or diagonals

In quasi macro-, micro- static indentation techniques the hardness of a material is evaluated using the measured diagonals of the residual indent, while in nanoindentations the indenting depth is monitored continuously during the loading-unloading cycle, but it is the final residual depth that’s used in determining the hardness. The hardness at a given load should be the same irrespective of whether the diagonal or depth of the residual indent is used in calculating the hardness i.e. it should be $\frac{d}{h} = \text{constant}$.

The geometric relationship between the diagonal and the depth for Vickers indenters is $d = 2h \tan 74.06^\circ = 7 h$. But in the indents the measured ratio of the diagonal to the depth, was found not to be a constant as shown in figure 5.8 (page 59)

A consequence of the ratio (d/h) being higher than 7 is that in the hardness calculated using the residual depth is greater than that calculated using the diagonal. In figure 5.9 the hardness evaluated using the measured diagonals and depth of residual indents for the WC 20 wt % Co sample is shown as an example.

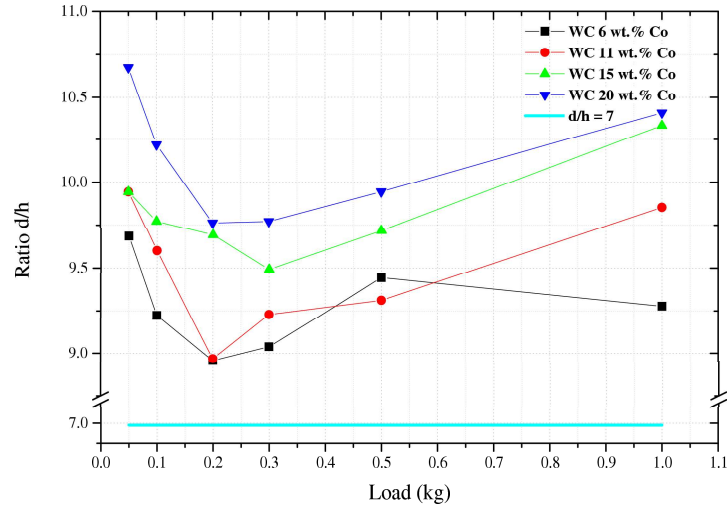


Figure 5.8: Change of d/h (ratio of residual diagonal to residual depth) for all the cemented carbides tested.

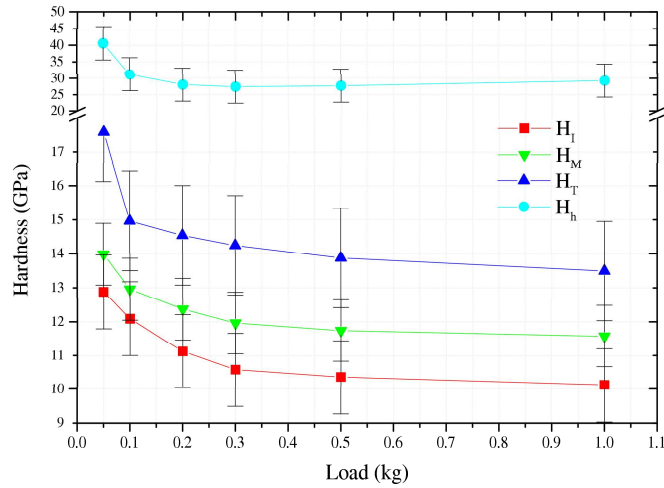


Figure 5.9: The variation of Vickers hardness due to measured diagonal and depth (H_h) of the residual indent at various indenting load for WC 20 wt.% Co sample. For comparison, hardness data for all the three diagonals (H_I , H_T and H_M) are also included.

5.4 Indentation size effect in cemented carbides

The Vickers macrohardness value for a given material is approximately constant i.e. load-independent. In the case of the WC-Co samples tested this value is

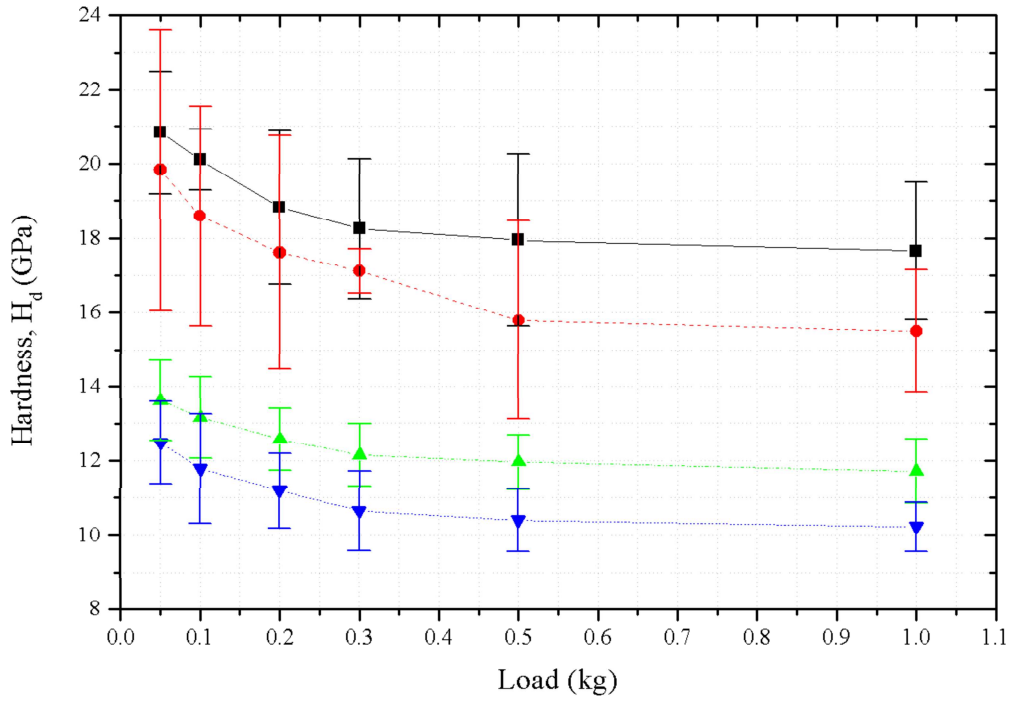


Figure 5.10: The hardness evaluated using the diagonal d_I as a function of load. The $\blacksquare$ is WC 6wt% Co, $\bullet$ is WC 11wt% Co, $\blacktriangle$ is WC 15wt% Co and $\blacktriangledown$ is WC 20wt% Co.

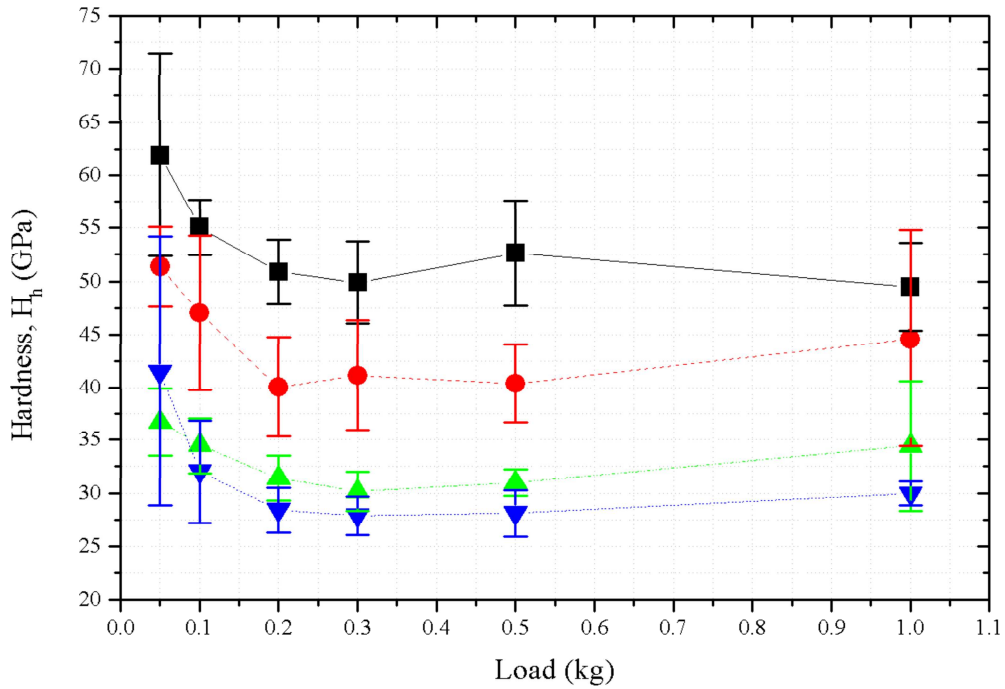


Figure 5.11: The hardness evaluated using the residual depth, h as a function of load. The $\blacksquare$ is WC 6wt% Co, $\bullet$ is WC 11wt% Co, $\blacktriangle$ is WC 15wt% Co and $\blacktriangledown$ is WC 20wt% Co.

attained from indenting loads $\geq 0.5 - 1$ kg.

Graphs of hardness as a function of applied indenting loads are given in figure 5.10 and figure 5.11 where the hardness is calculated using the diagonal d_I and the residual indent depth h respectively. In both cases, an increase in hardness with decreasing applied indenting load has been observed for all the four cemented carbides samples tested. Although the hardness in figure 5.11 is much higher than that observed in figure 5.10 nevertheless there is a significant increase in hardness with decreasing loads for the four samples.

5.5 Pileups

Pileups are defined as the material protruding around the indent that is higher than the "original" surface, with most of the pileups being along the sides of the indent and lesser along the corners of the indent. In figure 5.1 (page 50), the line profile along the diagonals shows the existence of pileups. Two issues pertaining to pileups are (i) shapes and size of pileups and (ii) the ratio of volume of pileups to the volume of residual indent. Both are dealt with in this section.

5.5.1 Shape and size of pileups

An AFM image consists of ample details of identifiable and distinguishable surface features of nanometer scale, which makes it possible to analyze the pileups of WC-Co. Typically, in the case of cemented carbides tested the pileups are of few micrometers in length and sub-micrometer in height.

In figure 5.1 of the line profile along the edges, the pileups are in the region between points T and I. Each line profile drawn across the indent and passing through point 'O', there are three points T, M and I that are defined (see Appendix Autoindent and figure 5.1).

Three terms are used to identify and defining the shape of the pileups; (i) The length of pileup L_{PU} is the sum of the projected distance between the points T to I and T' to I', (ii) The length L_{TM} is the sum of the projected length between T to M and T' to M' and (iii) The length L_{MI} is the sum of the projected length

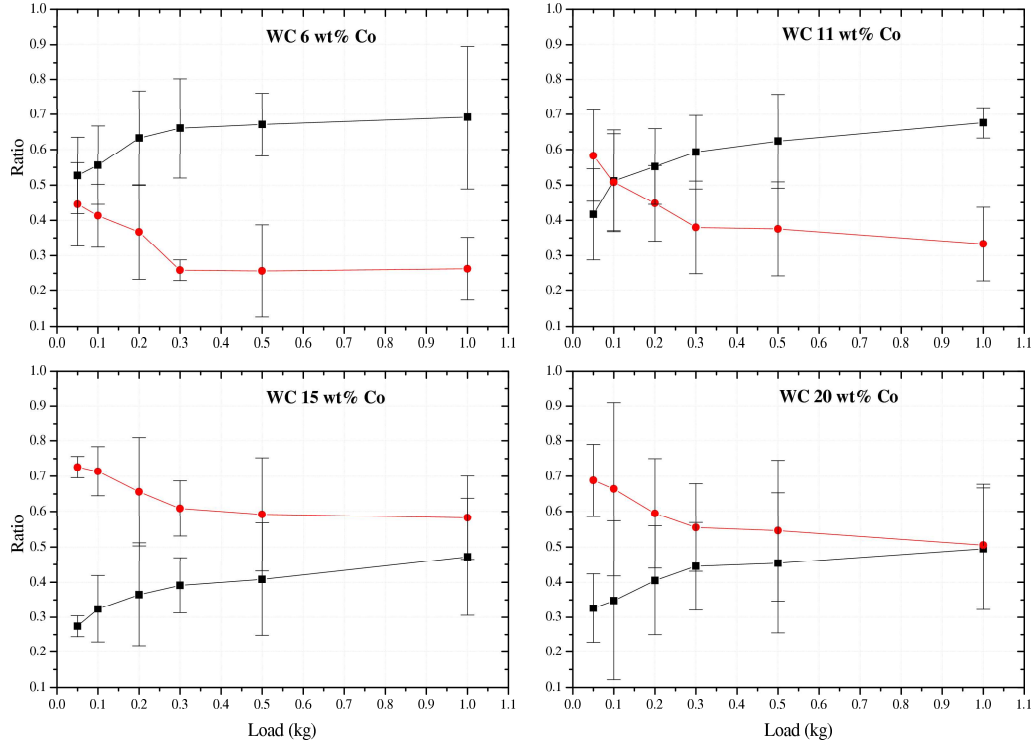


Figure 5.12: The solid squares corresponds to the ratio L_{MI} to L_{PU} , while the closed circles corresponds to the ratio L_{TM} to L_{PU} . The vertical axis in all the four graphs represents the ratio and the horizontal axis gives the applied indenting load.

between M to I and M' to I'. The ratio of the lengths L_{TM} and L_{MI} to L_{PU} for the four samples tested are given in figure 5.12 (page 62).

5.5.2 Volume of pileups and residual indent

Since the AFM images also contain height information it is possible to measure the volume of a given feature. The volume of residual indent V_I and volume of pileups V_{PU} as function of applied load is given in figure 5.13 (page 63). The calculations for the volume of residual indent and pileups are given in figure 4.7 and section 4.3.3. In figure 5.14 (page 63) the ratio of volume of residual indent to pileups are given for the four WC-Co samples tested.

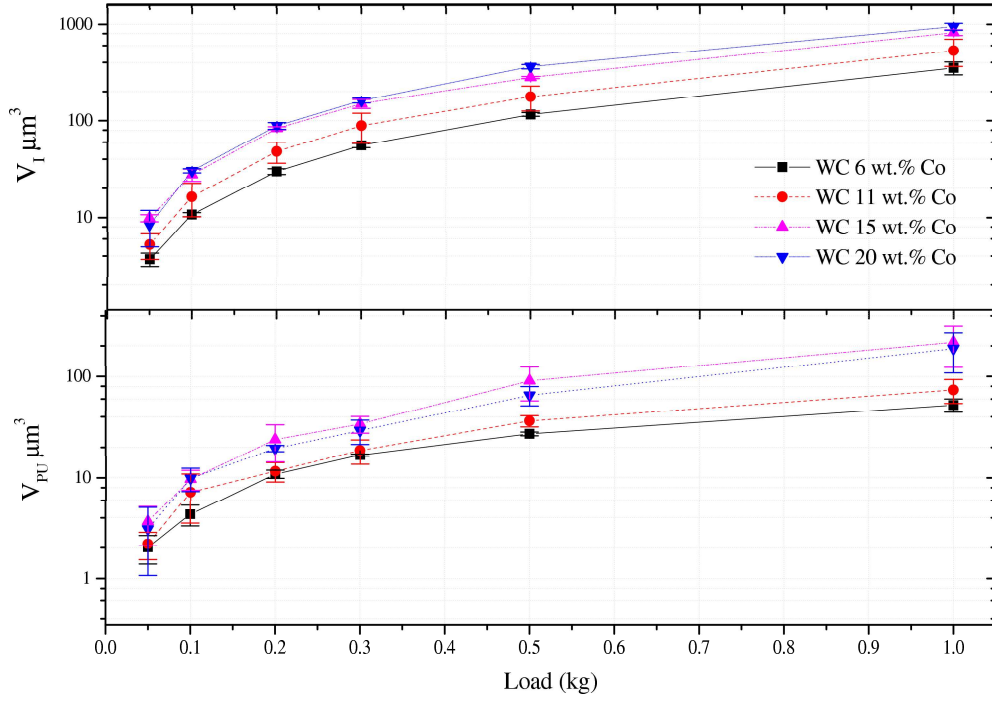


Figure 5.13: The volume of residual indent V_I and volume of pileups V_{PU} , top and bottom graphs respectively, as determined by the program AutoIndent.

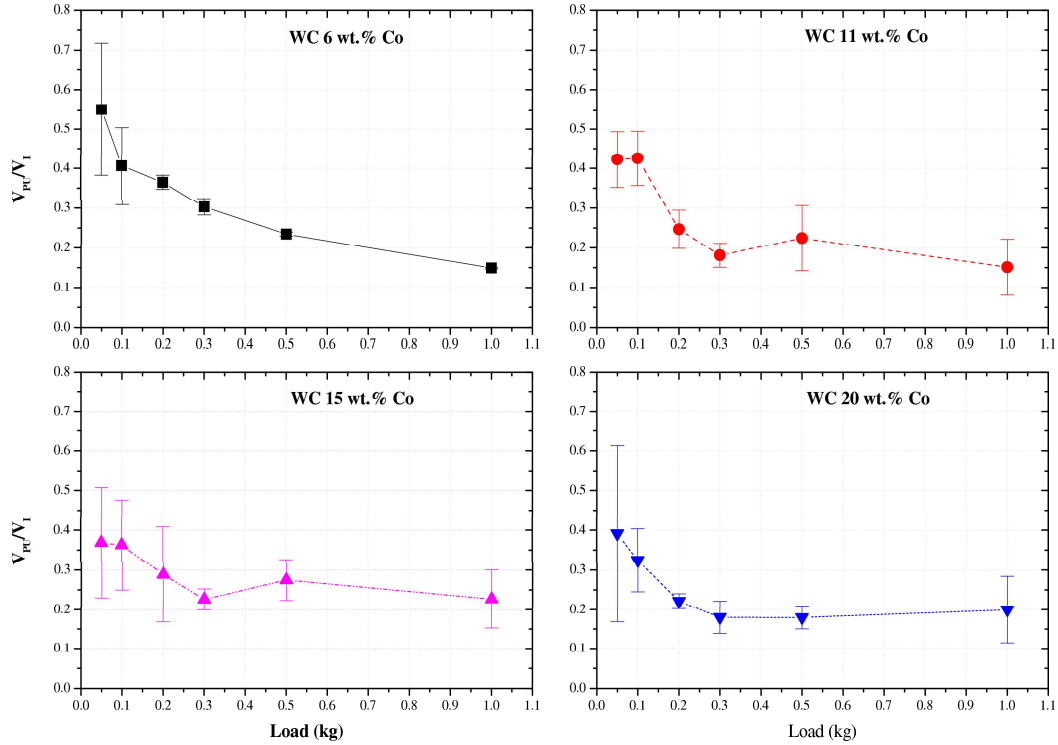


Figure 5.14: Ratio of volume of pileup to volume of residual indent V_{PU}/V_I .

Chapter 6

Discussion

The size dependence of the hardness of WC-Co at the micron scale was demonstrated in Chapter 5. In the present chapter attempts will be described to explain the observed size dependence (ISE) in terms of relationships among the parameters measured by AFM and the parameters calculated from the results of those measurements (table 5.1).

The first relationship that was explored was that between the ratio $(H/H_0)^2$ and $1/h$ (where H is the hardness of the material at a given load, H_0 the macroscopic hardness, and h is the residual depth of the indentation [Nix, 2002] at the load applied to measure H). Nix and Gao [Nix and Gao, 1998] predicted this relationship to be linear on the basis of a dislocation model that allowed them to estimate the density of the geometrically necessary dislocations (GND, see Chapter 2, section 2.2) which are generated under a loaded indenter to accommodate strain gradients. The relationship (reported again below) was derived to explain the ISE on a micron scale and was found to be linear in one-phase materials [Stelmashenko et al., 1993], [DeGuzman et al., 1993], and [Nix and Gao, 1998]. In the case of WC-Co the relationship was not expected to be necessarily linear, at least for the following reasons:

1. The parameters $\rho = \rho_S + \rho_G$ (dislocation density while ρ_S and ρ_G are the SSD density and GND density respectively), μ (shear modulus) and b (Burgers vector) which are well defined in one-phase materials are not clearly defined in two-phase materials, since they would be different in the two different phases;

2. The relationship does not consider interactions between dislocations and grain boundaries, which would be particularly important in a two-phase material;
3. The residual microstrains present in a two-phase materials are expected to affect the strain gradients generated by indentations.

According to Nix and Gao's theory the dependence of hardness on the depth of the indentation is given by the following relationship:

$$\left(\frac{H}{H_0}\right)^2 = 1 + \frac{h^*}{h} \quad (6.1)$$

where, as said above H and h are the measured hardness and the residual depth of indentation respectively, and H_0 is the hardness at the limit of infinite depth, while h^* is a characteristic length that depends on the shape of the indenter, the shear modulus and dislocation density through H_0 . The terms h^* and H_0 are given by

$$H_0 = 3\sqrt{3}\alpha\mu b(\rho_s)^{1/2} \quad (6.2)$$

and

$$h^* = \frac{81}{2}b\alpha^2\tan^2\theta\left(\frac{\mu}{H_0}\right)^2. \quad (6.3)$$

Therefore, according to Nix and Gao [Nix and Gao, 1998] a linear relationship exists between $(H/H_0)^2$ and $1/h$ which has been confirmed by other researchers ([Chechenin et al., 1997], [Elmustafa et al., 2000], [Qiu et al., 2001], [Swadener et al., 2002], [Feng and Nix, 2004], [Al-Rub and Voyiadjis, 2004], [Cao and Lu, 2005], [Durst et al., 2005], [Alkorta et al., 2006], [Balint et al., 2006], [Huang et al., 2006]).

However, an attempt to apply maximum strain gradient theory to the data in table 5.1 has provided unexpected insights into the ISE in WC-Co. At first the data in table 5.1 were substituted in equation 6.1 and plotted as shown in figure 6.1. The macrohardness H_0 for WC-Co grades was taken to be the hardness measured at a load of 1kg. It has been observed for macroindentation for the four WC-Co grades, the hardness for load > 1 kg to be almost constant or with negligible variation in hardness with load, by optical microscopy. Jia *et al.*[Jia et al., 1998]

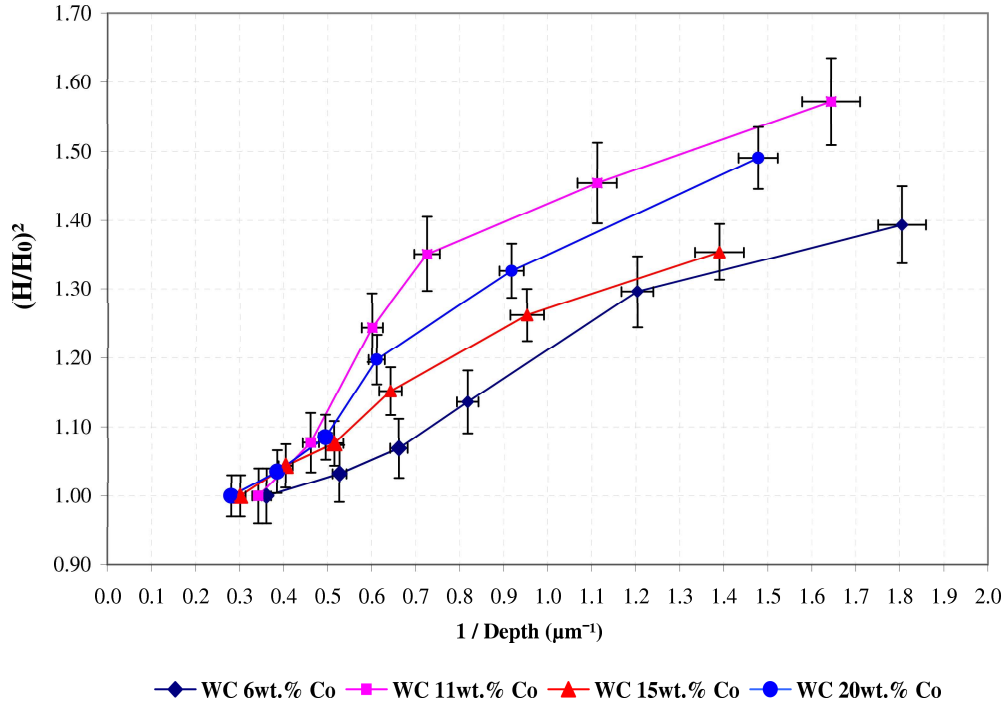


Figure 6.1: Nix and Gao model applied to the results from cemented carbides.

have also observed in case of cemented carbides WC-Co, the hardness is almost constant for indenting loads > 1 kg.

Nix and Gao [Nix and Gao, 1998] obtained straight lines (see figure 2.2 page 12) when plotting $(H/H_0)^2$ vs $1/h$ while the lines obtained for WC-Co are not straight lines (figure 6.1).

However, Nix and Gao equation 6.4 can be re-written as follows:

$$H^2 = H_0^2 + H_0^2 h^* \frac{1}{h}, \quad (6.4)$$

Again, on the basis of equation 6.4 when plotting H^2 vs. $1/h$ one should obtain a straight line for a one-phase material.

Figure 6.2 shows that when H^2 vs. $1/h$ was plotted for the WC-Co grades tested the curves obtained were “bilinear” i.e. appears to consist of two straight lines instead of one, similar to the curves obtained by Elmustafa *et al.* [Elmustafa et al., 2004] when applying the same model to α -brass, copper and aluminum.

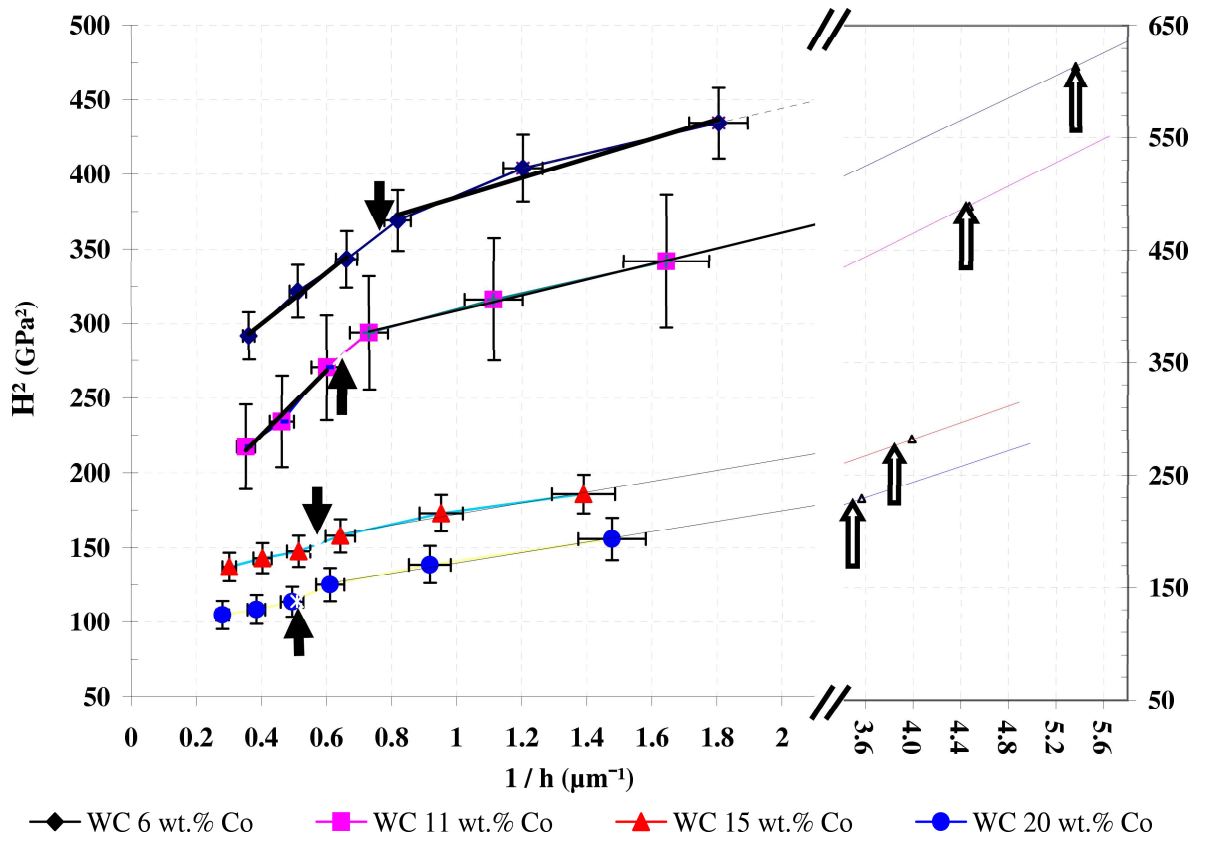


Figure 6.2: The square of the hardness as a function of the reciprocal of the depth of the Vickers indentation for samples of WC-Co with four different weight percentages of cobalt binder. The filled vertical arrows indicate the stages of indentation at which the depth h of the indentation is equal to the intrinsic characteristic length ζ of the sample; the open arrows indicate the stages at which the diagonal of the indentation is equal to the characteristic length.

From the data of H^2 vs $1/h$ (figure 6.2) it is observed that a sudden change in gradient occurs at a value of $1/h = 1/\zeta$ (see filled vertical arrows in figure 6.2) for all the WC-Co grades, which is related to the microstructure of WC-Co, as it has been found that ζ is approximately the sum of the WC grain size (λ) and the mean free path of Co binder (χ) in all grades. The term ζ is defined therefore as the "intrinsic characteristic length" of WC-Co, given by

$$\zeta = \lambda + \chi \quad (6.5)$$

Although Elmustafa and Stone [Elmustafa et al., 2000] had observed bilinear behaviour they did not identify a parameter that would account for the change in gradient from lower loads to higher loads. In the case of WC-Co, it has been possible to identify that the transition occurs at an "intrinsic characteristic length".

Henceforth, the data in figure 6.2 can be divided into two - for the case when $h > \zeta$ and when $h < \zeta$. The values of H_0 and h^* for each of the straight lines in (figure 6.2) using equation 6.4 were determined by linear regression fit with correlation coefficient of $\sim 97\%$. The obtained values are summarized in table 6.1, along with values of ζ , h_t , H_t and HV , where $\zeta = \lambda + \chi$ is the intrinsic characteristic length of WC-Co, λ the average WC grain size and χ the mean free path of Co, which were determined by linear analysis (table 3.1), H_t and h_t are the hardness and depth values respectively at the point of change in gradient, with HV being the macrohardness of the WC-Co samples measured experimentally.

Table 6.1: Values of H_0 and h^* for WC-Co for $h > \zeta$ and $h < \zeta$. H_t and h_t are the hardness and depth at $h = \lambda + \chi = \zeta$ respectively. The macrohardness HV and ζ is the intrinsic characteristic length of WC-Co (see table 3.1). h_t is the value obtained from figure 6.2 and $\zeta = \lambda + \chi$ is the value obtained from microstructure analysis.

Sample	$h > \zeta$		$h < \zeta$					
	H_0 (GPa)	h^* (μm)	H_0 (GPa)	h^* (μm)	H_t (GPa)	h_t (μm)	HV (GPa)	$\zeta = \lambda + \chi$ (μm)
WC 6wt.%Co	14.94	0.84	17.88	0.20	18.61	1.3	17	1.31
WC 11wt.%Co	11.80	1.54	16.01	0.21	16.62	1.41	15.5	1.57
WC 15wt.%Co	11.08	0.40	11.65	0.27	12.28	1.52	11.7	1.82
WC 20wt.%Co	9.62	0.45	10.25	0.33	10.67	1.69	10.7	2.17

Comparing the value of H_0 and HV for the two cases $h > \zeta$ and $h < \zeta$, it is observed, that for the case of $h < \zeta$, $H_0 \simeq HV$, for all the WC-Co samples, while for the case of $h > \zeta$, $H_0 < HV$. This suggests that Nix and Gao's equations 6.2, 6.3 apply to $h < \zeta$ but do not apply to $h > \zeta$. This observation can be related to the recent results by Abu Al-Rub [Abu Al-Rub, 2007] who found that the Nix and Gao's model applies to microindentation and not nanoindentation, however it requires further investigation.

From equation 6.3, the term h^* is inversely proportional to the square of the macrohardness H_0 , therefore it is expected that when the value of H_0 decreased the value of h^* increases. In case of WC-Co the macrohardness is known to decrease (table 3.1 and 6.1) with increasing cobalt content and for the case $h < \zeta$, the calculated value of h^* increases with decreasing H_0 (determined hardness) as cobalt content increases. However, for $h > \zeta$, the value of h^* decreases with increasing cobalt content, which further suggests that the Nix and Gao's model does not apply to these values, table 6.2 summarizes this information.

Table 6.2: The table summarizes the trends observed with respect to increasing cobalt content for WC-Co samples for the values of H_0 and h^* obtained in table 6.1.

	Cobalt content	$H_0 (= HV)$	h^* ($h^* \propto 1/H_0^2$)
Expected	increases	decreases	increases
$h < \zeta$	increases	decreases	increases
$h > \zeta$	increases	decreases	decreases

It is interesting to relate the diagonal of the indentations to the characteristic length ζ . Figure 6.3 shows that at all loads the length of the diagonal d of the indentation is approximately the same multiple of ζ for all grades, i.e.

$$d = \kappa\zeta = \kappa(\lambda + \chi) \quad (6.6)$$

with κ increasing with increasing load but having approximately the same value for all grades at each load. This supports the statement that $\zeta = \lambda + \chi$ is an intrinsic characteristic length of WC-Co. It must be noted that the intrinsic characteristic length ζ identified in this work does not correspond to the characteristic length h^* defined by others who have investigated the ISE in one-phase

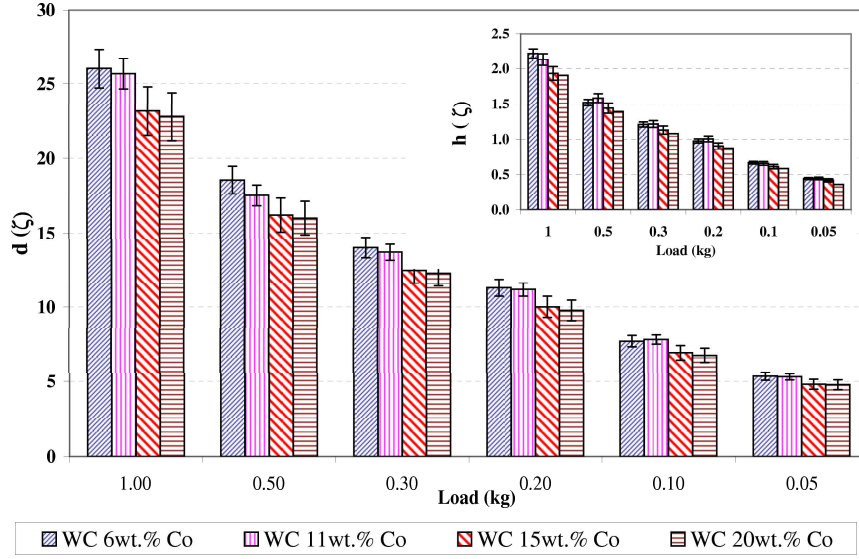


Figure 6.3: Diagonal (in ζ units) versus load. The insert graph shows the residual depth (in ζ units) versus load, for all the WC-Co grades.

materials [Nix and Gao, 1998], [Zong and Soboyejo, 2005], [Nix et al., 2007], [Swadener et al., 2002], [Elmustafa et al., 2000], [Feng and Nix, 2004].

The hardness H , can be expressed in terms of ζ

$$H = c \frac{L}{d^2} = c \frac{L}{\kappa^2 \zeta^2} = c \frac{L}{\kappa^2 (\lambda + \chi)^2} \quad (6.7)$$

where L indenting load, c depends on the indenter geometry ($c = 1.854$ for Vickers indenter), and κ a constant which increases with increasing load.

Alternately when the hardness H is expressed in terms of the depth, the hardness is given by

$$H = c' \frac{L}{\kappa'^2 (\lambda + \chi)^2}$$

where $c' \approx 24.5$ for Vickers indenters and κ' is a constant increasing with increasing load. Therefore the measurement of hardness is reduced to the measurement of κ (or κ'). But from the data in figure 6.3 it is possible to derive a parabolic relationship between κ and L so that for every ζ (i.e. for every grade of a given grain size and mean free path) once the load is know the hardness is know, without the need of measurements.

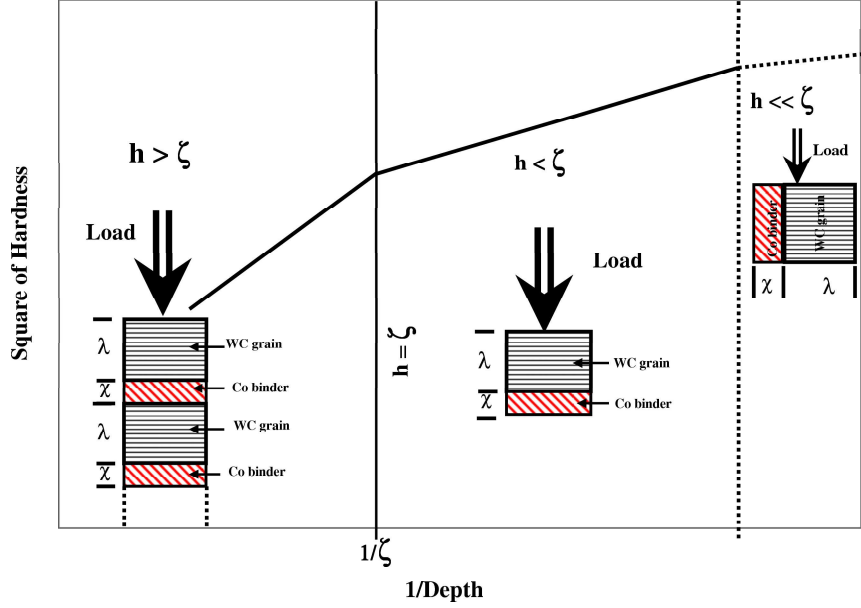


Figure 6.4: Schematics of square of hardness as a function of reciprocal of depth, relating the indentation depth to the microstructure of WC-Co. When $h > \zeta$ the indenter penetrates more than one WC grain and more than one Co layer, while when $h \leq \zeta$ the indentation penetrates only one WC grain and one Co layer. For the case, when $h \ll \zeta$ the indentation occurs predominantly in either the WC grain or in Co layer.

Figure 6.4 summarizes schematically the information acquired from the above analysis. It shows that when the indentation depth is $h \leq \zeta$ the plastic deformation due to the indentation is limited to a surface layer thickness of the order of ζ or less, while when $h > \zeta$ the thickness of the surface layer involved is of the order of more than one $\zeta = \lambda + \chi$ units.

Figure 6.4 shows schematically that by decreasing the indenting loads (and indentation depths) below the values tested in this investigation one would eventually test only single phases (either WC grain or a cobalt layer), i.e. the transition would occur from a two-phase to a one-phase material.

It must be noted that although the results of this investigation confirm that in materials exhibiting the ISE a constant material length scale can be defined and is related to microstructural characteristics [Nix and Gao, 1998], the intrinsic characteristic length ζ identified for WC-Co does not correspond to the charac-

teristic length h^* defined by others who have investigated the ISE in one-phase materials [Nix and Gao, 1998], [Zong and Soboyejo, 2005], [Nix et al., 2007], [Swadener et al., 2002], [Elmustafa et al., 2000], [Feng and Nix, 2004]. In fact, according to equation 6.1, h^* is equal to the indentation depth when $H^2 = 2H_0^2$ but table 6.1 shows that within the range of loads used in this investigation H never reached the value of $\sqrt{2}H_0$, thus in the case of WC-Co it is always $h^* \ll \zeta$.

The aims of the project have been achieved. However, it was hoped that the application of the Nix and Gao model to the results obtained from WC-Co would lead to an explanation of the ISE in these materials. Attempts to explain the bilinear graphs in figure 6.2 have proved unfruitful probably because the effects of interfaces and residual thermal stresses should be taken into account.

Chapter 7

Conclusions

The AutoIndent program that has been developed specifically to analyze indentations measured by atomic force microscopy, has allowed to measure systematically the diagonal, residual depth and angle along the diagonal and to determine the volume of residual indent and volume of the piled up material around the indent. By comparing the hardness values determined by means of AutoIndent program and by means of scanning electron and optical microscopy it has been possible to establish that the latter techniques measure the diagonal of indentations including the “piled-up” material at the indentations corners. This concurs with the measurements of [McElhaney et al., 1998], [Elmustafa et al., 2000] although no attempts have been made by either of the authors to explain why the pile-ups are included in the measurements of the diagonal (area), while an attempt has been made here to explain the unexpected observation. As far as other detailed observations are concerned, it has been found that the volume of the residual indent and the volume of the piled-up of the material around the indent are not equal, contrary to what was generally assumed, and that the piled-up material around the indentations has a different shape for the four WC-Co samples, and a change in shape of piled-up material is observed from WC 11 wt.% Co to WC 15 wt.% Co, which suggests a dependence on cobalt content.

Additionally, from measurements by atomic force microscopy and by means of the AutoIndent program the indentation size effect in WC-Co has been confirmed and it has been proved that microindentation in WC-Co recovers elastically when the diamond indenter is unloaded (appendix E). Recovery of the diagonal length, the indentation depth and the indentation angle have been observed, with higher

recovery in depth than in diagonal or angle. However, a correction factor has been derived that allows to compensate for the indentation size effect, i.e. allows to express the hardness as a constant at all indenting loads.

In an attempt to explain the indentation size effect in WC-Co, the data obtained by AFM have been fitted into a modified Nix and Gao model (equation 6.4 and 2.19), and the result is that the plot H^2 vs $1/h$ is bilinear (as opposed to the monolinear behaviour observed in one-phase materials) with the transition from the one gradient to the other at an indentation depth equal to $\zeta = \lambda + \chi$ (where λ is the mean grain size of WC and χ is the mean free path of Co) for all the WC-Co grades. On account of this result ζ has been identified as an intrinsic characteristic length of the material. It must be noted that this is the first direct experimental observation of an intrinsic characteristic length in a material.

Increasing cobalt content at any given load (L) the diagonal length (d) of the indentation increases, with increasing mean free path χ (and at constant WC grain size), but $d/(\lambda + \chi)$ remains constant, for all the four WC-Co grades tested as shown in figure 6.3. This allows to express the hardness H in terms of intrinsic characteristic length ζ

$$H = k \frac{L}{\psi^2 \zeta^2} = k \frac{L}{\psi^2 (\lambda + \chi)^2}$$

where k is a constant dependent on the indenter geometry and ψ is a constant for all cobalt contents at a constant grain size, but increases with increasing indenting load.

In conclusion, therefore, this work has unveiled many unknown aspects of the plastic behaviour of WC-Co when indented. Some of the aspects have been considered to be beyond the scope of the present project, therefore they need future investigations. The most important of these aspects are the following:

1. to explain why by scanning electron and optical microscopy the piled-up material outside the indentations is included in the measurement of the indentation diagonals
2. to explain the "bilinear" behaviour" of the plot H^2 vs $1/h$ for WC-Co, which contrasts with the monolinear behaviour in one-phase materials. This analysis will have to take into account the effect of residual stresses and interfaces on the plastic behaviour of the material.

3. to explore if the observed "bilinear" behaviour mentioned above is observed in other two-phase materials;
4. to extend the investigation (which has been limited to WC-Co grades of equal WC grain size and different cobalt contents) to grades of constant cobalt content and different grain sizes. It is important to assess if $\zeta = \lambda + \chi$ is still an intrinsic characteristic length of the material when the grain size is changed.
5. to investigate why the shape of the material piled-up around the indentations changes when going from low-Co to high-Co grades, since this may provide information on differences in plastic and/or elastic behaviour between high-Co and low-Co alloys.

Appendix A

Finite Element Analysis of the diamond indenter

The elastic strain of the diamond indenter induced during hardness tests was determined by finite element analysis for each of the samples and each indenting load [Dough, 2000]. A series of three-dimensional finite element (FE) stress analyses were carried out using the general purpose finite element code ABAQUS/Standard [Hibbitt and Sorensen, 1998]. The finite element analysis was carried out by FEAS¹ [Dough, 2000].

Table A.1 summaries the deflection of the diamond indenter's tip under each load. In table A.2, the diagonal deflection of the indenter under full-loads are given. Due to the elastic deformation of the diamond indenter's tip and the diagonal, the half-angle between the opposite edges of the indenter and along the diagonal also changes. This change is given in table A.3.

Table A.1: Tip deflection of Vickers' diamond indenter under full given load

Load (kg) (kg)	WC 6wt.% Co (μm)	WC 11wt.% Co (μm)	WC 15wt.% Co (μm)	WC 20wt.% Co (μm)
0.05	0.11	0.091	0.11	0.1
0.1	0.16	0.13	0.11	0.13
0.2	0.21	0.21	0.19	0.17
0.3	0.27	0.24	0.22	0.21
0.5	0.34	0.31	0.29	0.26
1	0.5	0.44	0.39	0.36

¹Finite Element Analysis Systems(FEAS) is a consulting company specializing in finite element analysis

Table A.2: The diagonal deflection of the diamond indenter under full load.

Load (kg) (kg)	WC 6wt.% Co (μm)	WC 11wt.% Co (μm)	WC 15wt.% Co (μm)	WC 20wt.% Co (μm)
0.05	0.02	0.02	0.05	0.05
0.1	0.076	0.063	0.052	0.06
0.2	0.066	0.1	0.1	0.06
0.3	0.091	0.1	0.111	0.1
0.5	0.095	0.13	0.14	0.14
1	0.27	0.21	0.2	0.15

Table A.3: Change in the half-angle along the edges of indenter due to tip and diagonal deflections.

Load (kg) (kg)	WC 6wt.% Co ($^{\circ}$)	WC 11wt.% Co ($^{\circ}$)	WC 15wt.% Co ($^{\circ}$)	WC 20wt.% Co ($^{\circ}$)
0.05	75.354	74.983	74.771	74.617
0.1	74.91	74.673	74.544	74.612
0.2	75.093	74.773	74.592	74.674
0.3	75.108	74.801	74.591	74.56
0.5	75.172	74.798	74.621	74.481
1	74.795	74.726	74.562	74.583

Appendix B

Measuring Indents by OM, SEM and AFM

Notations used in this appendix are related to figure B.1:

d_O diagonal measured by optical methods (bold solid lines)

d_S diagonal measured by SEM (bold dashes lines)

d_T diagonal measured by AFM between points T and T'

d_M diagonal measured by AFM between points M and M'

d_I diagonal measured by AFM between points I and I'

B.1 Indentation diagonal

Usually residual indents are measured by optical microscopy (OM) and for higher magnification they are often measured under a Scanning Electron Microscope (SEM). Different aspects of indentations have been subject of study by different electron microscope techniques [Motz et al., 2004], [Lloyd et al., 2005]. In some cases, in-situ indentations have been performed in a SEM [Gane and Bowden, 1968], [Stelmashenko et al., 1993] observing the behaviour of a material during indentation.

A limitation of both the techniques (OM and SEM) for measuring microindentation is their lateral resolution and depth of field resolution. The lateral resolution

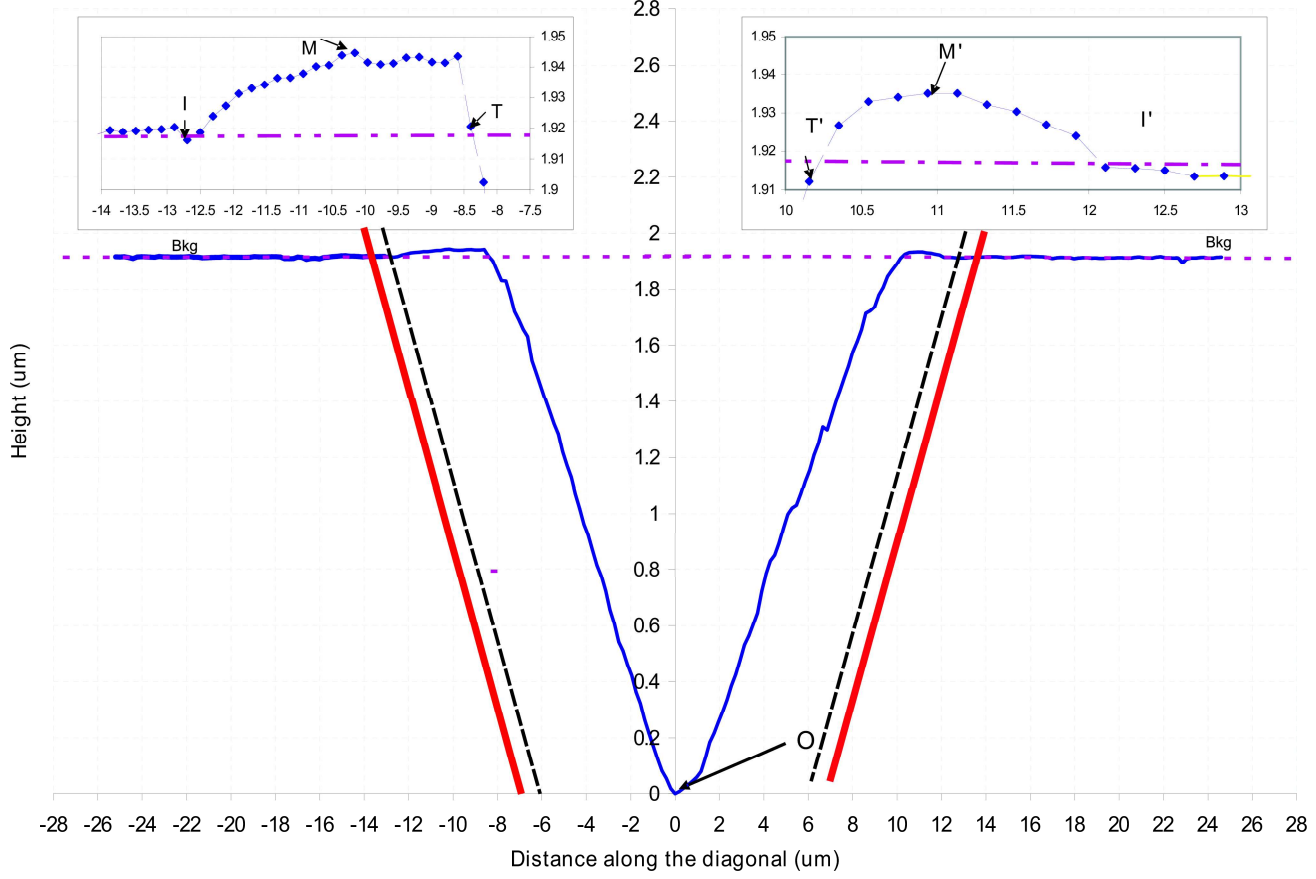


Figure B.1: Line profile along the opposite edges of a residual indent obtained by AutoIndent. The y-axis gives height with respect to the minimum point 'O'. In the main figure, the solid-blue line represents the line profile along the opposite edges of the residual indent and while the purple dotted horizontal line represents the 'original' surface. The points T, T', M, M', I and I' are defined in the text. The inserts shown are the section of the line profile (i.e. solid blue line) representing the pileups section magnified on either sides of the indent by the dashed blue lines. The solid red line represents the optical measurements while the dashed black line represents the SEM measurements.

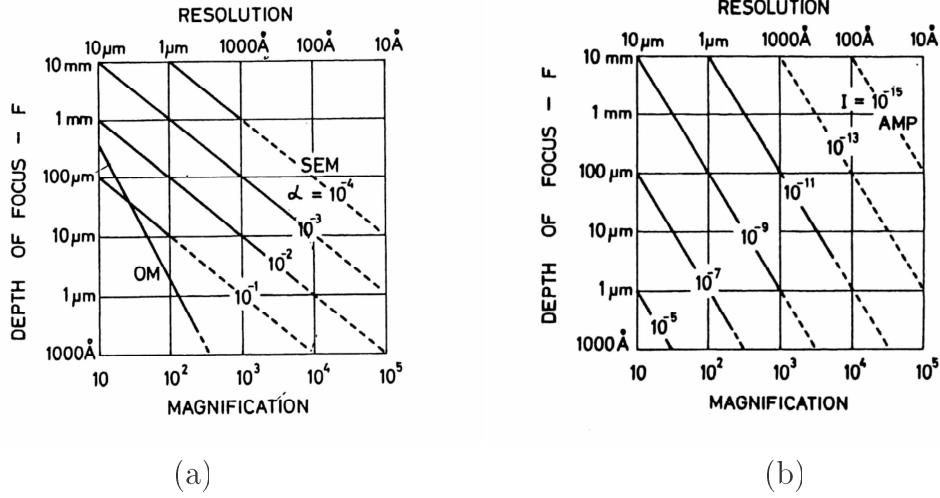


Figure B.2: The relationship between depth of focus F , resolution R and magnification (a) as a function of beam divergence angle α . (b) as a function of electron beam current [Booker, 1969].

for OM is of the order of the wavelength used (400 - 700 nm). During the measurements, the entire indent is brought into the field of view and focused, identifying the indent's fringes. Markers identifying the fringes due to the opposite corner of the indent are used for measuring the diagonal of the indent.

In the cemented carbides tested, the length of the diagonals of the microindentations (indenting loads $< 1\text{ kg}$) are in the $5 - 50\text{ }\mu\text{m}$ range. In figure B.5 (page 86) the SEM micrograph shows an entire residual indent within its field of view. The images were acquired at magnifications of $10^3 - 10^4\times$, with electron beam current of 10^{-10} A . Figure B.2 gives the relationship between the magnification, depth of focus, resolution, beam divergence and beam current for SEM. For the beam current range used and $10^3 - 10^4$ magnification the depth of focus is $10 - 1\text{ }\mu\text{m}$ with lateral resolution of $1000 - 100\text{\AA}$.

In OM and SEM the diagonals are measured from a two-dimensional representation (micrograph) of the residual indent, while in the case of the AFM the diagonals are measured from a three-dimensional image. Figure B.3 compares the diagonals of the residual indents measured by SEM and AFM with that measured by OM. The bold solid line of gradient -1 , represents limit of correlation between

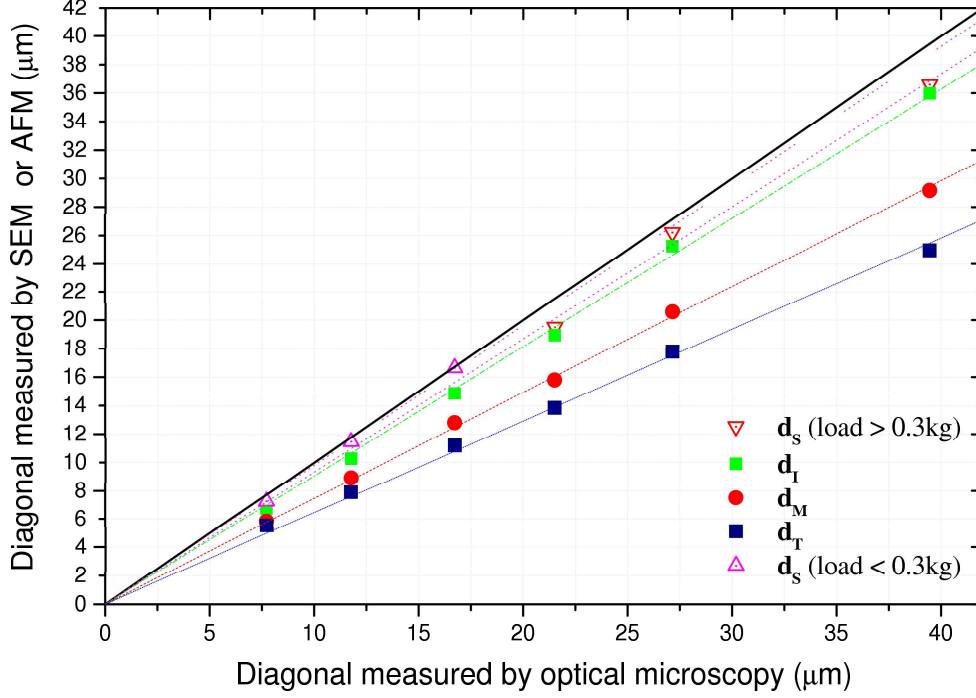


Figure B.3: Plot of diagonal measured by SEM and AFM against the diagonal measured by optical microscopy d_O . d_S , d_T , d_M and d_I have been defined under notations used. The values of d_O would lie on the solid line.

Table B.1: This table refers to the lines in figure B.3. The ratio between the diagonals measured by SEM and AFM and the diagonal measured by optical microscopy.

Diagonal	gradient (m)
d_S load $\geq 0,3\text{kg}$	0.934
d_I	0.907
d_M	0.746
d_T	0.646
d_S load $\leq 0,2\text{kg}$	0.982

Table B.2: Percent average difference in the diagonal values $\Delta d_{ij}/d_i\%$, $d_{ij} = d_i - d_j$ with $i = O, S, T, M, \text{ or } I$ and $j = O \text{ or } S$.

	d_O	d_S	d_I	d_M	d_T
$\Delta d_i/d_O$	-	5% ± 3	11% ± 2	25% ± 1	34% ± 3
$\Delta d_i/d_S$	5% ± 3	-	6% ± 4	20% ± 2	30% ± 3

the diagonal measured by OM and other techniques with lateral resolution higher than that of OM. It is noticed that there is a good agreement between the diagonals measured by OM and SEM for applied load ≤ 0.3 kg. The average difference between d_O and d_S is 5%. By contrast, the average difference between d_O and d_T is $\approx 35\%$. Table B.2 summarizes the percentage differences among the various diagonals d_O , d_S , d_I , d_M and d_T measured.

The differences in the diagonals measured by OM, SEM and AFM are due to (i) mechanisms by which an image is formed, (ii) lateral resolution, (iii) depth of field (or depth of focus) resolution. Optical images are influenced by wavelength (λ), interference (diffraction) between incident and reflected rays, medium and magnification at which the image is acquired. For an optical microscope, the resolution R is given by $R = \lambda/(N.A.) = \lambda/n \sin \phi$ where $N.A.$ is the numerical aperture of the lens, n reflective index of the medium and ϕ is the beam divergence. The SEM resolution [Booker, 1969] is dependent on: (a) electron beam spot size (b) electron beam spreading effects in specimen (c) signal to noise ratio (d) environmental factors such as stray fields, mechanical vibrations. The graphs given in figure B.2 show the relationship between depth of focus, resolution and beam divergence for OM and SEM (graph on the left), while the graph on the right shows the relationship between depth of focus, resolution and electron current in beam spot.

A comparison of the hardness evaluated from the data acquired by OM, SEM and AFM, shows that the hardness evaluated from AFM diagonal d_I is close to the OM and SEM evaluated hardness. This raises two issues; (i) Does the diagonal measured by SEM and OM include pileups? (ii) What are the consequences of using d_I (diagonal inclusive of the pileups) instead of d_T (diagonal excluding pileups)?

Most literature dealing with the comparison of SEM and AFM [VanHelleputte et al., 1995], [Castle and Zhdan, 1997] focuses on lateral dimensions of nanometer scale and are often concerned with protruding features. In the present case, the features of interest are depressions in the sample with shallow angle with respect to the ‘original’ surface (i.e. the indentation) and with features around these shallow features (i.e. the pileups) of nanometer height.

In SEM micrographs the residual indents are identified by observing the differences in contrast of the images. SEM micrographs of cemented carbides consist of two features: the darker regions which represent the cobalt phase of the material, and the gray (brighter) regions are the WC grains (figure 3.1). The residual indentations in SEM micrographs consist of three regions within a “squarish” feature corresponding to the residual indent: (a) The darker region is due to the secondary electrons emitted from the indent surface not facing the detector; (b) the brighter region indicative of a large amount of emitted secondary electrons being collected by the detector, i.e. a region of the residual indent facing the detector; (c) the edges of the residual indent which are boundaries between dark and bright regions.

The diagonals are measured by measuring the distance between opposite corner points of the “squarish” region of the residual indent. The corner points are along the edges, where the uniform contrast of the original surface starts. However we are now faced with the issue of $d_S \simeq d_I > d_T$ and $H_T \gg H_I \simeq H_S$. In models in continuum mechanics ([Hill, 1950], [Bishop et al., 1945] [Tabor, 1951] [Johnson, 1970]) on the behaviour of material during indentation it is usually assumed that the diagonals of the residual indents exclude the pileups.

It is observed by AFM that there are pileups present all around the residual indents, a feature that was not observed in the SEM, even with the sample tilted at 82.5° (figure B.4). Goncalves [Goncalves, 1992] using a differential interference contrast (DIC) microscope and Talysurf observed pileups around residual indents in cemented carbide for macroindentations. Although DIC provides a qualitative image in vertical direction, it has a depth of field resolution of a few nanometers and lateral resolution similar to OM. SEM micrographs of residual indent within field of view were acquired at magnifications $10^3 - 10^4\times$ and of lateral resolutions of $1000 - 100\text{\AA}$ with depth of focus of $10 - 1\mu\text{m}$. The two techniques DIC and SEM complement each other with higher depth of field and higher lateral resolutions

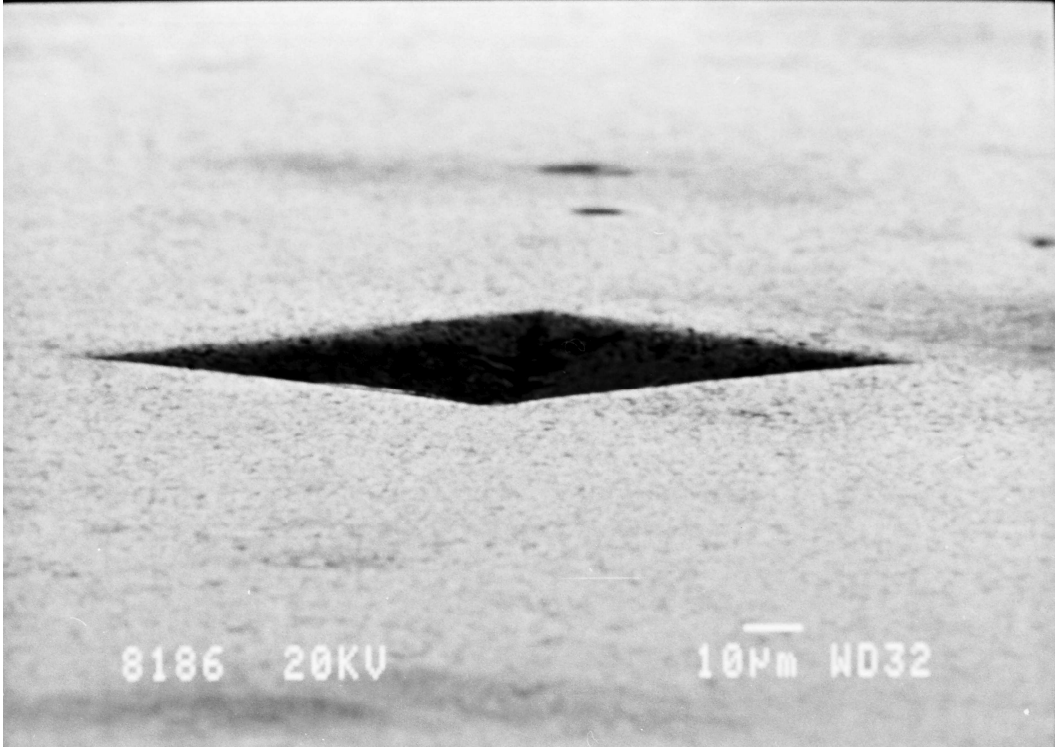


Figure B.4: SEM image of indent titled at 82.5° . The residual indent was produced at a 10 kg indenting load. The lack of contrast along the sides and around the corners seems to indicate absence of pileups.

respectively.

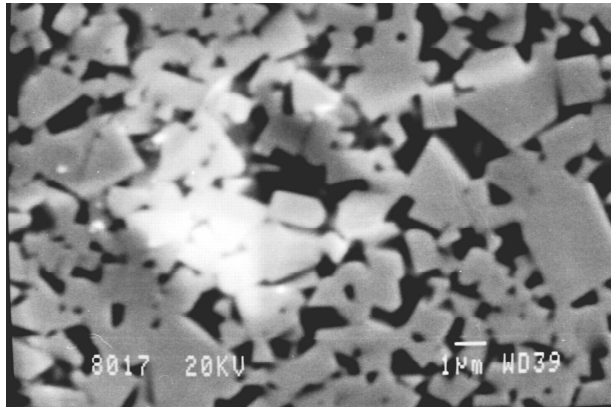
A qualitative comparison of SEM and AFM images for indenting loads of 1kg and 50g is shown in figure B.5. Both SEM and AFM images give good resolution images of the 1kg residual indent. While in the case 50g indent, the indent is well defined in the AFM image but not in the SEM image. This must be due to the limitations of SEM, in the depth of field resolution as well as the lateral resolution, because (i) the features of the 50g indent are of the order of $< 1\mu\text{m}$ and the pileups are $< 0.1\mu\text{m}$ which is of the order of the depth of field resolution of SEM.

SEM and AFM images for indenting loads of 1kg and 50g as shown in figure B.5. The height of the pile-ups of the material around the corner of the indent is of the order of a few nanometers as measured from the AFM images. In case of the SEM image, the variation in the gray contrast around the corners of the indents can not significantly distinguish between a protrusion and a depression of a few

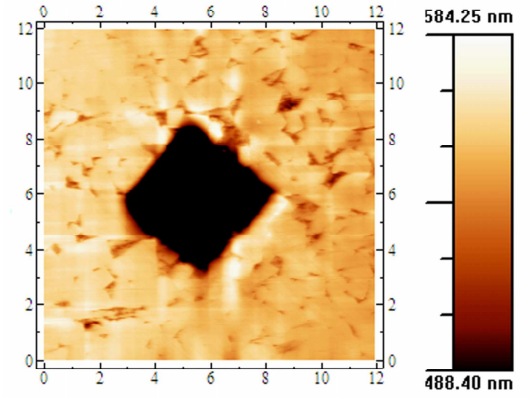
nanometers in vertical heights or depth. Thus, it is not possible to determine by SEM if the measurements of the diagonal includes or excludes pileups. However, the measurements of the diagonal by SEM are almost equal to that of the diagonal d_I measured by AFM.

Thus for the purpose of evaluating the hardness for the cemented carbides the diagonal d_I which includes the pile-ups of the material along the diagonal line profile (see figure B.1) is used in the present work. [McElhaney et al., 1998], [Elmustafa et al., 2000] have similarly shown that a correlation of 1 is obtained between SEM and optical microscopy measurements if the pile-ups of the material around the indents are taken into accounts for the evaluation of the hardness. However, if the diagonal d_T (i.e. does not contain any contribution from the pile-ups) is used for evaluating the hardness then the hardness obtained is almost twice its macro-hardness.

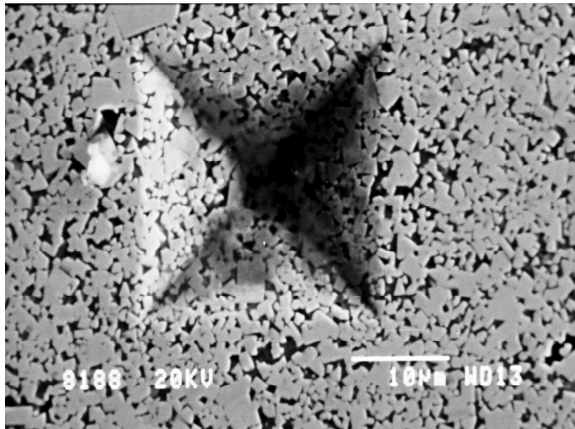
Using the program AutoIndent for the analysis of the AFM residual indent images three sets of pairs of points are distinguishable in each diagonal line profiles. The points T and M (figure B.1) seem to be intuitively relevant in defining the indent, however the hardness evaluated from the diagonals d_T and d_M is far greater than the macrohardness of the samples. The diagonal between the points II' provides hardness values at 1 kg indenting loads closets to the expected macro-hardness of the samples. Hence, for comparing the microindentation hardness with that of the macrohardness, the diagonal measured by AFM includes the pile-ups of the material along the diagonal line profile d_I .



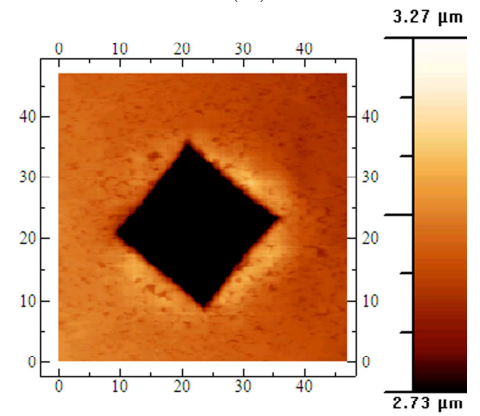
(a)



(b)



(c)



(d)

Figure B.5: SEM and AFM images of indentations for applied indenting loads of 50g (top two images) and 1kg (bottom two images). The SEM images are on the left while the AFM images are on the right. The x-y axis of the AFM images size are in μm . The gray scale adjacent to the AFM images is for display only, the actual AFM image the darkest region is always 0 nm.

Appendix C

Confocal stereology image of residual indent

The confocal stereology image of the residual indent on a replica of diffraction grating, acquired in transmission mode¹. The change in shape of the indentation when the indenter is unloaded is clearly visible in a replica material while is not as clearly visible in WC-Co, this work demonstrates what happens in WC-Co on a smaller scale. This stereo image provides visualization of the residual indent. Further magnification of the image allows for an in-depth observation of pattern of indent, around the surface as well as below the indent. Although, its difficult to quantify the density of pixelation or infer thereof the increase in material displaced under the indent (see figure C.1).

If one wear the stereology glasses with the green filter on the right eye and red filter on the left eye - the indent appears to be protruding from the surface of the plane of the page. Points of interest in this image are:

1. The edges of the residual indents are curved.
2. The sides of the residual indents are curved along both the length of the sides as well as along their width.
3. The corners of the indent are not well defined, and extend into the “original” surface.

¹Acknowledge Caroline Lalkan, Microscopy Unit, WITS for taking the photos

4. In the given stereology image the extent to which the edges of the indent extend into the surface are clearly visible if the stereology glasses are worn with red filter on the right eye.

The curved surfaces and edges are indicative of the non linear relaxation of the material during unloading process. The edge of the indent is define (formed) when two adjacent planes (sides) of the residual indent meet. The interface between the edge of the indent and the original surface is difficult to define, since the curved surface of the adjacent sides of the indent form a decreasing acute angle between the normal to the curved surfaces along the edges going from the apex of the indent to the original surface.

On wearing the stereology glasses with the green filter on the left eye and red on the right eye, the indent seems to be embedded into the page. In this case the extent of the residual indent becomes distinct, and the above mentioned changed in the acute angle becomes apparent.

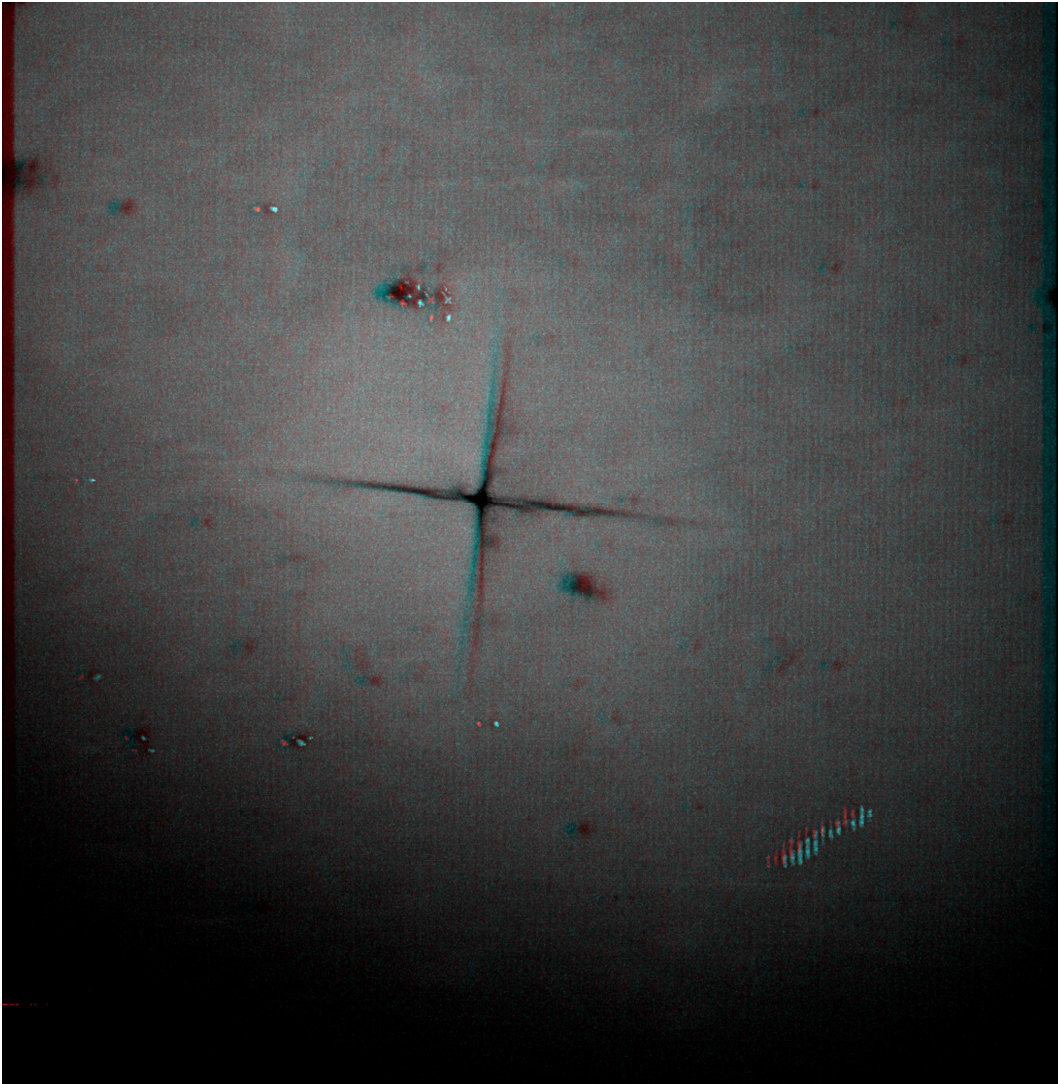


Figure C.1: Confocal Stereology image of residual indent of a replica diffraction grating.

Appendix D

Program AutoIndent

Structure of Program:

1. Read HDF file
2. Identify minimum point in the image
3. Identify the corners of the indent
4. Draw line profile between the minimum point and the corner points extending the line till the edge of the image
5. Combine line profiles to give the line profile along the edges of the indents, that is along the diagonal
6. Identify the points O, T, M, I and T', M', I'
7. Determine the Volume of the residual indent and Volume of the pile-ups of the material around the indent
8. Calculate, diagonal, depth, angle, volume and send the results to an output file.

AutoDig

```
function [volume,volpileup,truediag,truez,truevol,  
trueangpt,truedz,scansiz,  
zcal,B,trueside,truesidez,truesideangpt,truesidedz] = autodig(m)
```

```
%function [volume,volpileup,truediag,truez,truevol,diaglen,maxB,
%      trueangpt,truedz,bkgh, scansiz, zcal,B,truesside,truessidez,
%      truessideangpt,truessidedz,XX1,YY1,XX2,YY2,dR1,dR2] = autodig(m)
%
% This program is used to calculate the diagonal, volume, angle and depth
% of the micro-indentation as measured by the AFM.
% 1) PSI file is read using the program READPSI - file required READPSI.M
% 2) Center (apex of the pyramid) and the corner points are determined to
% determine the points for the diagonal PYRPTS.M or PYRLPTS.M
% 3) Line profile across the diagonal points is drawn. LINEPTS.M
% 4) From the line profile the following information is obtained.
% I) TP: Turning point i.e points between min and left or
% right side of % the profile ceases to be linear
% ii) Max:Maximum point of the pile up
% iii) IPU or IP: Including Pile up point
% FILE USED are LNPTSLN.M and LNPTSRN.M
% ..LN evaluated the points to the left of the min point
% ..RN evaluated the points to the right of the min point
% NB: This program allows input from the user!!!
% 5) Once the points for TP,Max and EP have been determined the Volume of
% indentation and volume of the pile up is determine. VOLAREA.M
% 6) The angle between the TP & Min, Max & Min, IPU-Max,Max-TP
% is determined
% LINEANG.M
%
% 7) Steps 3, 4 and 6 are repeated for the center of the sides of indentations
% SIDEVAL.M
%
% 8) volume,volpileup,
% truediag,truez,truevol, trueangpt,truedz,
% scansiz, zcal,
```

```
%      trueside,truesidez,truesideangpt,truesidedz,
%      Results are sent to a text file named data.
%
%   TESTED
%   created by Sanjiv Shrivastava
%   5 Nov. 1999

if nargin < 1
    [B,pyr,pyrln,0,scansiz,zcal,ptsl,ptmin,bkgh]= pyrpts;
else
    [B,pyr,pyrln,0,scansiz,zcal,ptsl,ptmin,bkgh]= pyrlpts;
end

disp('line profile across the diagonal')
[dR1,dZ1,dR2,dZ2,XX1,YY1,XX2,YY2,dmval] =
linepts(B,ptsl,ptmin,scansiz,zcal,bkgh);

figure, zoom
%subplot(2,1,1)
ptsleft1 = lnptsln(dR1,dZ1,scansiz,zcal) ; digl1 =ptsleft1;
ptsright1 =lnptsrn(dR1,dZ1,scansiz,zcal) ; digr1=ptsright1;

disp('in-pu, max of pu, turning pts, ex-pu, min pts')

ipur = abs( digl1(1,1)-digr1(1,5));
mpr = abs(digl1(1,2)-digr1(1,4));
tpr = abs(digl1(1,3)-digr1(1,3));
epur =abs( digl1(1,4)-digr1(1,2));

ipuz = mean([digl1(2,1) digr1(2,5)]);
mpz = mean([digl1(2,2) digr1(2,4)]);
tpz = mean([digl1(2,3) digr1(2,3)]);
```

```

epuz =mean([digl1(2,4) digr1(2,2)]);

distr1=[ipur mpr tpr epur] distz1=[ipuz mpz tpz epuz]

%subplot(2,1,2)
figure zoom
% Diagonal 2
ptsleft2 = lnptsln(dR2,dZ2,scansiz,zcal) ;
digl2=ptsleft2;
ptsright2=lnptsrn(dR2,dZ2,scansiz,zcal) ;
digr2=ptsright2;
disp('turning pts, max of pu, in-pu, in-pu true, ex-pu, min pts')

disp('in-pu, max of pu, turning pts, ex-pu, min pts')

ipur = abs( digl2(1,1)-digr2(1,5));
mpr = abs(digl2(1,2)-digr2(1,4));
tpr = abs(digl2(1,3)-digr2(1,3));
epur = abs( digl2(1,4)-digr2(1,2));

ipuz = mean([digl2(2,1) digr2(2,5)]);
mpz = mean([digl2(2,2) digr2(2,4)]);
tpz = mean([digr2(2,3) digl2(2,3)]);
epuz =mean([digl2(2,4) digr2(2,2)]);

distr2=[ipur mpr tpr epur] distz2=[ipuz mpz tpz epuz]

disp('in-pu, max of pu, turning pts, ex-pu, min pts')
diaglen = [ptsleft1; ptsright1 ; 1 1 1 1 1; ptsleft2; ptsright2]
disp('in-pu, max of pu, turning pts, ex-pu, min pts')
truediag=([distr1;distr2])
truez=([distz1;distz2])

```

```

figure [volume,volumepu] =
volarea(B,diaglen,XX1,YY1,XX2,YY2,scansiz,truez);

truevolume=volume(1:2,:)*(scansiz*scansiz / (256*256));
truevolnnz =volume(3,:);
truevol=[truevolume;truevolnnz];
[trueangpt,truedz]=lineang(B,diaglen,truediag,truez,scansiz,zcal);
trueangpt=trueangpt; truedz=truedz;

disp('gvol,Tvolipu,svol,nntrueipu,tzbkg')
%volpileup=volumepu;
volpileup=[volumepu(1:3)*(scansiz*scansiz /
(256*256)),volumepu(4),volumepu(5)];

im = input('Would you like to save the results! Y/N [Y]: ', 's') ;
    if im == 'y'
        disp('Saving results to file DATA')
        save data volume volpileup truediag truez truevol trueangpt truedz
        scansiz zcal trueside truesidez truesideangpt truesidedz -ascii
    elseif im == 'n'
        disp('Saving results to file DATA2')
        save data2 volume volpileup truediag truez truevol trueangpt truedz
        scansiz zcal trueside truesidez truesideangpt truesidedz -ascii
    end

```

ReadPSI

```

function [A,fname,imwidth, imlength, scansiz, zscal] =
readpsi(Asize)

%function [A,fname,imwidth, imlength, scansiz, zscal] = readpsiH(Asize)
%
% READPSI.m is the new file which reads in the scansize, and

```

```
%      zcalbration values from the psi header.
%      [A,fname,imwidth, imlength, scansiz, zscal] = readpsiH (Asize)
%      Temp Asize is used to specify the size of the image to
%      be read. This feature will be disabled.
%      A      is the psi-data matrix generated.
%      fname      name of the file read
%      imwidth     size of the scan i.e 256 or 512
%      imlength    size of the scan i.e 256 or 512
%      scansiz     Size of the image scan
%      zcal        z-calibration
%
%
%      Program:      Sanjiv Shrivastava
%      Date:         19 October 1998
%      Date modified:  -----
%
%      NOTE: Asize has been disabled. *****
%function [A,fname,imwidth, imlength, scansiz, zscal] = readpsiH (Asize)


%[fid,message] = fopen(uigetfile('c:\spmdata\wcindent\*.hdf',
%      'Topo File'),'rb')
%[fid,message] = fopen(uigetfile('c:\spmdata\*.hdf','Topo
File'),'rb') fname = fopen(fid)


positon = ftell(fid); status = fseek(fid,978,0); ps978x =
fread(fid,[1,1], 'short'); ps980y = fread(fid,[1,1], 'short');


status = fseek(fid,994-982,0);


ps994 = fread(fid,[1,1], 'float32'); ps998 = fread(fid,[1,1],
'float32');
```



```

status = fseek(fid,1042-1002,0);

ps1042 = fread(fid,[1,1], 'float32');

positon = ftell(fid); imwidth = ps978x ; imlength = ps980y ;

status = fseek(fid,16384-1046,0);

A = fread(fid,[imlength,imwidth],'short'); A = A'; status =
fclose(fid);

scansiz = ps994

zscal = ps1042

disp('imwidth, imlength, scansiz, zscal') disp('No. of pixels, Scan
Size (um), Z-calibration (um)') disp([imwidth, scansiz, zscal])

%function [A,fname] = readpsi(Asize)
% THIS WAS THE OLD FORMAT OF THE FILES READ BY MATLAB.
%
%[fid,message] = fopen(uigetfile('*.hdf','Topo File'),'rb')
%fname = fopen(fid)
%positon = ftell(fid);
%status = fseek(fid,16384,0);
%positon = ftell(fid);
%A = fread(fid,[Asize,Asize],'short');
%A = fread(fid,[256,256],'short');
%A = A';
%status = fclose(fid);

```

```
% OLD VERSION
%ps978 = fread(fid,[1,1], 'uchar');
%ps979= fread(fid,[1,1], 'uchar');
%ps980= fread(fid,[1,1], 'uchar');
%ps981= fread(fid,[1,1], 'uchar');

% OLD VERSION
% ps994 = fread(fid,[1,1], 'uchar');
% ps995 = fread(fid,[1,1], 'uchar');
% ps996 = fread(fid,[1,1], 'uchar');
% ps997 = fread(fid,[1,1], 'uchar');

% OLD VERSION

% ps1042 = fread(fid,[1,1], 'uchar');
% ps1043 = fread(fid,[1,1], 'uchar');
% ps1044 = fread(fid,[1,1], 'uchar');
% ps1045 = fread(fid,[1,1], 'uchar');

%A = fread(fid,[Asize,Asize], 'short');

% m3=   ps994;
% m2=   ps995;
% e2m1= ps996;
% se1=  ps997;
% sign=(se1/128);
% expon=((se1*2)+(e2m1/128)-127);
% m1=e2m1-((e2m1/128)*128);
% mant=1.+m1/128.+m2/(128.*256.)+m3/(128.*256.*256.);
% scansiz=mant*exp(expon*log(2.))*1000. ;
% scansiz = scansiz/1000;
```

```
%m3= ps1042;
%m2= ps1043;
%e2m1= ps1044;
%se1= ps1045;
%sign=(se1/128);
%expon=((se1*2)+(e2m1/128)-127);
%m1=e2m1-((e2m1/128)*128);
%mant=1.+m1/128.+m2/(128.*256.)+m3/(128.*256.*256.);
%zscal=mant*exp(expon*log(2.))*1000.;
%zscal =zscal/1000
```

PyrPts

```
function [B,pyr,pyrln,0,scansiz,zcal,pts,ptmin,bkgh]= pyrpts

% INPUT: [A,fname,imwidth, imlength, scansiz, zscal] = readpsi
% OUTPUT: [B,pyr,pyrln,0,scansiz,zcal,pts,bkgh]= pyrpts
%          grad = gradient from apex

% The program works as follows:
% Followin input is required by the user
%          1)      Image to be analysed
%          2)      Scansize
%          3)      z-calibration factor
%          4)      no. of points per bin
%          [NB: if entire image is to be used then dif =1]
%
% Procedure:
%          1) Apex of the indentation is found. [point 0]
%          2) If dif ~= 1, then matrix 'B' is created
%          3) Distance between the apex and points [i,j] is calculated
%              and matrix 'dist' is created giving the distance in um.
%          4) Difference in height from the apex is calculated
%              matrix 'difz' is created the given values are in um
```

```
%      5) Radius between apex and the point angrd == distance sqrt(x^2 %

%      This routine is used to find the diagonal line
%
%      1)      Find the peak of the pyramid
%      2)      Create matrix Bpyr 80% of height
%      3)      Create background height 95%
%      4)      Compare matrix Bdist versus Bpyr
%      5)      Find max(max(Bdist)) and draw a line between apex
%                  of Pyramid and

[A,fname,imwidth, imlength, scansiz, zscal] = readpsi ;

scansize = scansiz    ; zcal = zscal    ; xscale = scansize/imlength ;
yscale = scansize/imwidth ;

minA = min(min(A)) maxA=max(max(A))
% point O: apex of the pyramid is given by
[ox,oy] = find(A == minA) ; O = [ox,oy,A(ox,oy)] ;

A = (A-min(minA)) * zcal; B =A; O=O; ox= O(1,1); oy=O(1,2);

maxB = max(max(B)) ; minB = min(min(B)) ; meanB = mean(mean(B));
maxmeanB = meanB/maxB

hp = input(' set threshold for the pyramid') ;

if hp > 1
    h2 = maxmeanB - 0.15 ;
elseif hp == []
    h2 = maxmeanB - 0.15 ;
else
```

```
h2 = hp ;
end h2=h2;

hr = input(' set Background threshold') ;

hrt = maxmeanB + 0.09;

if hr > 1 & hrt > 1
    h3 = 1.00 - 0.05 ;
elseif hr == []
    h3 = maxmeanB - 0.07 ;
else
    h3 = hr ;
end h3=h3;

% approximates the pyramid(indentation)

Bpyr = (B < maxB*h2);

pyr = zeros(size(Bpyr));

[i,j] = find(Bpyr == 1); if i ~=[] & j ~=[] V =[i,j];
    [a,b] = size(V);
    for n =1:a
pyr(V(n,1),V(n,2)) =B(V(n,1),V(n,2)); end end

% used to calculate the background height
%

Brem = (B > maxB*h3);
```

```

bkg = zeros(size(Brem)); [i,j] = find(Brem == 1); if i ~= [] & j ~=
[] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
bkg(V(n,1),V(n,2)) = B(V(n,1),V(n,2)); end end

bkgn = nnz(bkg); bkgh = sum(sum(bkg))/bkgn ;

pyrln = edge(pyr);

[i,j] = find(pyrln == 1); v = [i,j]; V = [v(:,2), v(:,1)];
% This gets the co-ordinate position in the matrix V

mini = find(V(:,1) == min(V(:,1))) ; maxi = find(V(:,1) ==
max(V(:,1))) ; minj = find(V(:,2) == min(V(:,2))) ; maxj = find(V(:,2)
== max(V(:,2))) ;

% Translate the above results into the image matrix points
%           V(minX,:)

if length(mini) > 1 ptmini = mean(V(mini,:)); else ptmini =
V(mini,:) ; end ptA = floor(ptmini);

if length(minj) > 1 ptminj = mean(V(minj,:)); else ptminj =
V(minj,:) ; end ptB = floor(ptminj);

if length(maxi) > 1 ptmaxi = mean(V(maxi,:)); else ptmaxi =
V(maxi,:) ; end ptC = floor(ptmaxi);

if length(maxj) > 1 ptmaxj = mean(V(maxj,:)); else ptmaxj =
V(maxj,:) ; end ptD = floor(ptmaxj);

```

```

ptmin=[ox,oy]

pts = [ptA ptB ptC ptD]

oyl = ones([1 256])*oy; oxl = ones([1 256])*ox;

figure hold on spy(pyrln) hold on
plot(oxl,','')
plot(oyl,(1:256),','')
plot(ptA(1,1),ptA(1,2),'*g') plot(ptB(1,1),ptB(1,2),'*c')
plot(ptC(1,1),ptC(1,2),'*r') plot(ptD(1,1),ptD(1,2),'*y') hold off
zoom

PyrMtx

function [pyr,pyrh,pyrln,pyred,pyredh,bkgh]=
pyrmtx(B,ang,dist,0,hr,hp)
%
%      This routine is used to find the diagonal line
%
%      1)      Find the peak of the pyramid
%      2)      Create matrix Bpyr 80% of height
%      3)      Create background height 95%
%      4)      Compare matrix Bdist versus Bpyr
%      5)      Find max(max(Bdist)) and draw a line between apex of
%                  Pyramid
%
%
%
B =B; ang=ang; dist =dist;
%angrd=angrd;
%angxy=angxy;
%angz=angz;

```

```

0=0; ox= 0(1,1); oy=0(1,2); h2=hp; h3=hr;

% approximates the pyramid(indentation)
maxB = max(max(B)) ; minB = min(min(B)) ; meanB = mean(mean(B));
maxmeanB = meanB/maxB;

if h2 > 1
    h2 = maxmeanB - 0.15 ;
else
    h2 = hp ;
end

Bpyr = (B < maxB*h2);

pyr = zeros(size(Bpyr)); pyrh = zeros(size(Bpyr)); pyrang =
zeros(size(Bpyr)); [i,j] = find(Bpyr == 1); if i ~=[] & j ~=[] V
=[i,j];
    [a,b] = size(V);
    for n =1:a
pyr(V(n,1),V(n,2)) =dist(V(n,1),V(n,2)); pyrh(V(n,1),V(n,2))
=B(V(n,1),V(n,2));
%pyrang(V(n,1),V(n,2)) =ang(V(n,1),V(n,2));
end end

% used to calculate the background height
%
if h3>1
    h3 = maxmeanB + .07;
else
    h3=hr ;
end

Brem = (B > maxB*h3);

```



```

bkg = zeros(size(Brem)); [i,j] = find(Brem == 1); if i ~= [] & j ~=
[] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
bkg(V(n,1),V(n,2)) = B(V(n,1),V(n,2)); end end bkg_n = nnz(bkg); bkg_h
= sum(sum(bkg))/bkg_n ;

% in order to find the opposite ends of the diagonal
% the image is split into 4 quadrants

quad1 = pyr(1:ox, 1:oy);
quad2 = pyr(1:ox, oy:256);
quad3 = pyr(ox:256, oy:256);
quad4 = pyr(ox:256, 1:oy);

maxq1 =max(max(pyr(1:ox , 1:oy) ) ) ; maxq2 =max(max(pyr(1:ox ,
oy:256) ) ) ; maxq3 =max(max(pyr(ox:256 , oy:256) ) ) ; maxq4
=max(max(pyr(ox:256 , 1:oy) ) ) ;

[q1x,q1y] = find(quad1 == maxq1) ; [q2x,q2y] = find(quad2 ==
maxq2) ; [q3x,q3y] = find(quad3 == maxq3) ; [q4x,q4y] = find(quad4
== maxq4 ) ;

q1xy = [q1x,q1y]; q2xy = [q2x,q2y+oy]; q3xy = [q3x+ox,q3y+oy]; q4xy
= [q4x+ox,q4y];

qpts = [q1xy;q2xy;q3xy;q4xy] ;

pyrln = edge(pyr);

pyred = zeros(size(pyr)); pyredh = zeros(size(pyr));
%pyredang = zeros(size(pyr));

```

```

[i,j] = find(pyrln == 1); if i ~=[] & j ~=[] V = [i,j];
    [a,b] = size(V);
    for n =1:a
pyred(V(n,1),V(n,2)) =dist(V(n,1),V(n,2)); pyredh(V(n,1),V(n,2))
=B(V(n,1),V(n,2));
%pyredang(V(n,1),V(n,2)) =ang(V(n,1),V(n,2));
end end

%*****
%   Using edge detection to find the diagonal points
%
%   This routine looks at the distance between the point
%       and apex

maxqed1 =max(max(pyred(1:ox , 1:oy) ) ); maxqed2 =max(max(pyred(1:ox
, oy:256) ) ); maxqed3 =max(max(pyred(ox:256 , oy:256) ) ); maxqed4
=max(max(pyred(ox:256 , 1:oy) ) );

[qed1x,qed1y] = find(pyred == maxqed1) ; [qed2x,qed2y] = find(pyred
== maxqed2) ; [qed3x,qed3y] = find(pyred == maxqed3) ; [qed4x,qed4y]
= find(pyred == maxqed4) ;

qed1xy = [qed1x,qed1y]; qed2xy = [qed2x,qed2y+oy]; qed3xy =
[qed3x+ox,qed3y+oy]; qed4xy = [qed4x+ox,qed4y];

qedpts = [qed1xy;qed2xy;qed3xy;qed4xy];

%*****

oyl = ones([1 256])*oy; oyl = ones([1 256])*ox;

```

```
figure spy(pyrln) hold on plot(oxl,'-') plot(oyl,(1:256),'-') hold
off
```

NewDiag

```
function [R1,Z1,R2,Z2,XX1,YY1,XX2,YY2] =
newdiag(B,pts,scansiz,zcal,bkgh,ptsmin)

B = B; ang=ang;
%pts = getpts

ptsA = pts(1,:); ptsB = pts(2,:); ptsC = pts(3,:); ptsD = pts(4,:);
ptmin =ptmin;

d1pts = [ptsA ; ptmin; ptsC]; d2pts = [ptsB ; ptmin; ptsD]; V1 =
d1pts; V2 = d2pts;

d1=polyfit(V1(:,1),V1(:,2),1) ; d2=polyfit(V2(:,1),V2(:,2),1);

x1 = V1(:,1); y1 = V1(:,2); f1=polyval(d1,x1); x2 = V2(:,1); y2 =
V2(:,2); f2=polyval(d2,x2);

DXX1 = abs(ptsA(1,1) - ptsC(1,1)); DYY1 = abs(ptsA(1,2) -
ptsC(1,2));

DXX2 = abs(ptsB(1,1) - ptsD(1,1)); DYY2 = abs(ptsB(1,2) -
ptsD(1,2));

if DXX1 >DYY1 disp('evaluating pts for line 1 in the x dir') DXX1;
    XX1 = (1:length(B));
    YY1 = size(XX1) ;
    for n=1:length(B) ;
        YY1(1,n) = d1(1)*n + d1(2) ;
```

```

        if YY1(1,n) <= 1 ;
            YY1(1,n) = 1 ;
        elseif YY1(1,n) >= 256 ;
            YY1(1,n) = 256 ;
        else
            YY1(1,n) = YY1(1,n) ;
        end
    end
    YY1 ;
        max(max(YY1));
else disp('evaluating pts for line 1 in the y dir') DYY1;
    YY2=(1:length(B));
    XX2 = size(YY2);
    for n=1:length(B)
        XX2(1,n) = (n - d1(2))/d1(1) ;
        if XX2(1,n) <= 1
            XX2(1,n) = 1 ;
        elseif XX2(1,n) >= 256
            XX2(1,n) = 256 ;
        else
            XX2(1,n) = XX2(1,n) ;
        end
    end
    end
    XX2 ;
    max(max(XX2)) ;
    XX1 = XX2 ;
    YY1 = YY2 ;
end
end

DXX2 = abs(ptsB(1,1) - ptsD(1,1)); DYY2 = abs(ptsB(1,2) -
ptsD(1,2));

```

```

if DXX2 >DYY2
    XX2 = (1:length(B));
    YY2 = size(XX2) ;
    for n=1:length(B) ;
        YY2(1,n) = d2(1)*n + d2(2) ;
        if YY2(1,n) <= 1 ;
            YY2(1,n) = 1 ;
        elseif YY2(1,n) >= 256 ;
            YY2(1,n) = 256 ;
        else
            YY2(1,n) = YY2(1,n) ;
        end
    end
    YY2 ;
    max(max(YY2))
else
    YY2=(1:length(B));
    XX2 = size(YY2);
    for n=1:length(B)
%   YY2(1,n) = d2(1)*n + d2(2) ;
        XX2(1,n) = (n - d2(2))/d2(1) ;
        if XX2(1,n) <= 1
            XX2(1,n) = 1 ;
        elseif XX2(1,n) >= 256
            XX2(1,n) = 256 ;
        else
            XX2(1,n) = XX2(1,n) ;
        end
    end
    XX2 ;
    max(max(XX2))
end

```

```

hold on plot(XX1,YY1,'.',XX2,YY2,'.') hold off

%
%   This routine is used to draw the plot
%   of distance vs height i.e matrix (B)
%

DX1 = XX1 -XX1(1,1); DY1 = YY1 -YY1(1,1); DX2 = XX2 -XX2(1,1); DY2 =
YY2 -YY2(1,1);

DX12 = DX1 .* DX1 ; DY12 = DY1 .* DY1 ; DX22 = DX2 .* DX2 ; DY22 =
DY2 .* DY2 ;

R1 = sqrt(DX12 +DY12); Z1 = size(R1) ; for i = 1:length(B)
    xx(i) = round(XX1(1,i));
    yy(i) = round(YY1(1,i));
    z1 = B(xx(i),yy(i));
Z1(1,i) = z1; end

R2 = sqrt(DX22 +DY22); Z2 = size(R2) ; for i = 1:length(B)
    xx(i) = round(XX2(1,i));
    yy(i) = round(YY2(1,i));
    z2 = B(xx(i),yy(i));
Z2(1,i) = z2; end

figure plot(R1,Z1,'g.') hold on plot(R2,Z2,'r-.')
```

LnptsL

```

function [ptsleft] = lnptsl(dR1,dZ1,scansiz,zcal);
```

```

dR1 =dR1; dZ1 = dZ1; scansiz=scansiz; zcal=zcal;

dr = dR1 * (scansiz/256); dz= dZ1;

%plots the entire line profile
plot(dr,dz,'oy')

% x = in = distance ; y = ij = height
% [in,jn] === gives the apex position.

[in,jn] = find(dz == min(min(dz)));

if length(jn) >1 jn =ceil(mean(jn)); end jn=jn;

hold on
% plots the min point (apex)
plot(dr(in,jn),dz(in,jn),'*g')

% gives the line profile left to apex

llpz = dz(1:jn) ; llpr = dr(1:jn);
% plots the selected line profile from apex to left
hold on plot(llpr,llpz,'.r')

% this finds the max of the pileup.

% THIS ROUTINE IS USED FOR FINDING THE GRADIENT AND THE
% DIRECTIONAL TANGENT
% Reference:
% Estimation of curvature and tangent direction by median
% filtered difference
% by; J matar, Z Shao & J Kittler

```

```
% 8 th Int. Conf., ICIAP'95, San Remo, Italy, Sept. 95
% LNCS 974, Image Analysis and Processing
% Page 83 -- 87

%
% STEP 1: Z and d are converted into polar co-ordinates
% 2: Plot (r vs theta)
% 3. derivate of theta
% 4. Plot derivate of theta vs r
% 5. determine changes from -ve to +ve or 0
% 6. find turning point
% 7. if (6) > 1; follow direction of tangent method
% 8. Determine the theta for
%
%NB: for the left side of the profile the apex is choosen to be (0,0)

lenpz=length(llpz);

for i=1:lenpz
    llpzs(i) = llpz(lenpz - i +1) - min(llpz);
% to change the origin (0,0) co-ordinates
end llpzs ;

for i=1:lenpz llprs(i) = llpr(lenpz-i +1) - max(llpr); end
llprs= abs(llprs); % because llpzs takes the value the llprs(1,1) ~= 0)
% hence llprs - max(llpr) such that llprs(1,1) == 0

% to convert from co- to polar we should
% subtract the origin llpzs - min(llpzs)
gradzs = (llpzs) ./ (llprs ); theta= 90 - atan(gradzs)*180/pi;

radi = sqrt(llpzs .* llpzs + llprs .* llprs); radius = abs(radi -
radi(1,1));
```



```

%figure
%plot(radius,theta,'*g')

% second derivative with respect to theta

[i,j] = find(llpr == 0); if i ~= [] & j ~= [] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
llpr(V(n,1),V(n,2)) = eps; end end

[i,j] = find(llpz == 0); if i ~= [] & j ~= [] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
llpz(V(n,1),V(n,2)) = eps; end end

zrgrad = llpr ./ llpz ; thetazr= 90 - atan(zrgrad)*180/pi;
%zrgrad2 = zrgrad(2:length(llpr)) ./ llpz(2:length(llpr)) ;
zrgrad2 = zrgrad ./ llpz ;

%figure
%plot(radi(2:length(llpr)),zrgrad2)

[irl] = find(zrgrad2 == min(zrgrad2));

if length(irl) > 1 irl == ceil(mean(irl)); end irl=irl;

%irl = irl-1;
leftr= radi(irl)

jtl = jn-irl+1 ;

```

```

hold on plot(dr(jtl),dz(jtl),'*c')

llpbkgz = dz(1:jtl) ; llpbkgr = dr(1:jtl);
% plot pileup + bkg
hold on plot(llpbkgr,llpbkgz,'-g')

% this finds the max of the pileup.

[iml] = find(llpz == max(max(llpz)));

if length(iml) >1 emlpt =abs(jtl-impl); eml=find(emlpt ==
min(emlpt)); jml = impl(eml); else jml = impl ; end

hold on plot(dr(jml),dz(jml),'*r')

% evaluates the bkg there are 4 methods of bkg subtraction
% bkg1 most appropriate method will be to use the mean of the llpbkgz

llpmz = llpz(1:jml); for i=1:jml llpmzr(i)= max(llpmz)
-llpmz(jml-i+1) ; end llpmzr;

llpmr = llpr(1:jml); for i=1:jml
llpmrr(i)=llpmr(jml-i+1)-max(llpmr); end llpmrr = abs(llpmrr);

radiip = sqrt(llpmrr .* llpmrr + llpmzr .* llpmzr); zrg= llpmrr ./
llpmzr ;
mzrg = radiip ./ zrg ;%./ radiip ;
mzrg2= mzrg ./ zrg ; iimpl = find(mzrg2 == max(mzrg2(2:jml)));
jimpl=jml-iimpl+1;

%*****

```

```

%lnbkgz = floor(0.5 .* length(llpbkgz)) ;

lnbkgz = floor(0.7 .* jml) ; bkg1 = mean(llpbkgz(1:lnbkgz)); bkg1min
= min(llpbkgz(1:jml)) ;

difbkg1 =abs(llpbkgz - bkg1); [bj] = find(difbkg1 == min(difbkg1));
if length(bj) > 1 bj = floor(mean(bj)); end bj=bj; [jipl] =
find(llpz == llpbkgz(bj) ); jipl = max(jipl);

% jipl -- inclusive of pile up using the mean
% jnpl -- " minimum

[jnpl] = find(llpbkgz(1:jml) == bkg1min);
% plots the background subtraction point
hold on plot(llpr(jipl),llpz(jipl),'*m')
plot(llpr,bkg1*ones(size(llpz)),'-m')
plot(llpr(jiml),llpz(jiml),'*c')
plot(llpr,bkg1min*ones(size(llpz)),'-r')

% NB conditions have to be set in order to choose which of the above
% three methods should be used as bkg

% find the point between max(pile up) and min(apex) == bkg
% to calculate the diagonal excluding the pile up

difbkl = abs(llpz(jml:jn) - bkg1); [i,j] = find(difbkl ==
min(difbkl)) ;
jepl = j +jml -1; % jml and not jtl

if length(jepl) >1 eppt =abs(jtl-jepl); ep=find(eppt == min(eppt));
jepl = jepl(ep); else jepl = jepl end

```

```

% plots the point excluding the pile up
hold on plot(llpr(jepl),llpz(jepl),'*g')

if length(jt1) <1 dltr = 'err' dltz = 'err' dltr = 0 ; dltz = 0 ;
else
dltr = llpr(jt1);    % turning pt of indent
dltz = llpz(jt1);    % turning pt of indent
end

if length(jml) <1 dlmr = 'err' dlmz = 'err' dlmr = 0; dlmz = 0; else
dlmr = llpr(jml);    % maximum of indentation
dlmz = llpz(jml);    % maxima of  indentation
end

if length(jipl) <1 dlipur = 'err' dlipuz = 'err' dlipur = 0 ; dlipuz
= 0 ; else
dlipur = llpr(jipl);    % includes pile up
dlipuz = llpz(jipl);    % includes pile up
end

if length(jiml) <1 dlimpur = 'err' dlimpuz = 'err' dlimpur = 0;
dlimpuz = 0; else
dlimpur = llpr(jiml);    % includes pile up true value
dlimpuz = llpz(jiml);    % includes pile up true value
end

if length(jepl) <1 dlepur = 'err' dlepuz = 'err' dlepur = 0; dlepuz
= 0; else
dlepur = llpr(jepl);    % excludes pile up
dlepuz = llpz(jepl);    % excludes pile up
end

```

```

if length(jn) <1 drmin = 'err' dzmin = 'err' drmin = 0; dzmin = 0;
else drmin = dr(in,jn); dzmin = dz(in,jn) ; end

disp('ptsleft = dltr    dlmr    dlipur dlimpur dlepur ');

ptsleft = [dltr dlmr dlipur dlimpur dlepur drmin;
           dltz dlmz dlipuz dlimpuz dlepuz dzmin] ;

% THIS CONCLUDES THE ROUTINE FOR IDENTIFYING 3 POINTS TO THE LEFT
%      OF THE APEX

% *****

LnptsR

function [ptsright] = lnptsr(dR1,dZ1,scansiz,zcal);

dR1 =dR1; dZ1 = dZ1; scansiz=scansiz; zcal=zcal;

dr = dR1 * (scansiz/256); dz= dZ1;

%figure
% plots the entire line profile
hold on plot(dr,dz,'-y')

[in,jn] = find(dz == min(min(dz)));

if length(jn) >1 jn =ceil(mean(jn)); end jn=jn;

hold on
% plots the min point (apex)
plot(dr(in,jn),dz(in,jn),'+g')
```

```
% gives the line profile left to apex
lndz=length(dz); rlpz = dz(jn:lndz) ; rlpr = dr(jn:lndz);
% plots the selected line profile from apex to left
hold on plot(rlpr,rlpz,'-c')

% this finds the max of the pileup.

% THIS ROUTINE IS USED FOR FINDING THE GRADIENT AND THE
% DIRECTIONAL TANGENT
% Reference:
% Estimation of curvature and tangent direction by median
% filtered difference
% by; J matar, Z Shao & J Kittler
% 8 th Int. Conf., ICIAP'95, San Remo, Italy, Sept. 95
% LNCS 974, Image Analysis and Processing
% Page 83 -- 87

%
% STEP 1: Z and d are converted into polar co-ordinates
% 2: Plot (r vs theta)
% 3. derivate of theta
% 4. Plot derivate of theta vs r
% 5. determine changes from -ve to +ve or 0
% 6. find turning point
% 7. if (6) > 1; follow direction of tangent method
% 8. Determine the theta for
%

%NB: for the left side of the profile the apex is choosen to be (0,0)

lenpz=length(rlpz);

rlpzr = rlpz - min(rlpz); % to change the origin (0,0) co-ordinates
```

```

rlpr = rlpr - min(rlpr);
rlpr = abs(rlpr); % because rlpzr takes the value the rlpr(1,1) ~= 0)
                % hence rlpr - max(rlpr) such that rlpr(1,1) == 0

% to convert from co- to polar we should subtract the origin llpzr - min(llpzr)
%gradzr = (llpzr) ./ (llpr );
%theta= 90 - atan(gradzr)*180/pi;

radi = sqrt(rlpzr .* rlpzr + rlpr .* rlpr);
radius = abs(radi - radi(1,1)); %

% second derivative with respect to theta

[i,j] = find(rlpr == 0); if i ~= [] & j ~= [] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
rlpr(V(n,1),V(n,2)) = eps; end end

[i,j] = find(rlpzr == 0); if i ~= [] & j ~= [] V = [i,j];
    [a,b] = size(V);
    for n = 1:a
rlpzr(V(n,1),V(n,2)) = eps; end end

zrgrad = rlpr ./ rlpzr; thetazr= 90 - atan(zrgrad)*180/pi;
zrgrad2 = zrgrad(2:length(rlpr)) ./ rlpzr(2:length(rlpr)) ;

[irl] = find(zrgrad2 == min(zrgrad2));

if length(irl) > 1 irl == ceil(mean(irl)); end irl=irl;

irl = irl + 1 ; rightr= radi(irl) ;

```

```

jtr = jn + irl -1 ; % Turning pt on the dz vs dr line profile

%point of the original line will be given by (length(llpz) + irl)
% TURNING POINT OF THE INDENTATION AS GIVEN BY d2r/d2theta
% rlpz(jn + irl); rlpr(jn + irl);

hold on plot(dr(jtr ),dz(jtr),'*c')

% this finds the max of the pileup.

[emr] = find(rlpz == max(max(rlpz)));

if length(emr) >1 emrpt =abs(jtr-emr); mr=find(emrpt == min(emrpt));
imr = emr(mr); else imr = emr ; end

jmr = jn+imr-1;

hold on plot(dr(jmr),dz(jmr),'*r')

rlpbkgz = dz(jtr:lndz); rlpbkgr = dr(jtr:lndz);

% plot pileup + bkg
plot(rlpbkgr,rlpbkgz,'-g')

% evaluates the bkg there are 4 methods of bkg subtraction
% bkg1 most appropriate method will be to use the mean of the llpbkgz

rlpmz = dz(jmr:lndz); % by using dr we eliminate the reindexing error
rln = lndz -jmr +1; for i=1:rln rlpzmzr(i)= max(rlpmz) -rlpmz(i) ;
end rlpzmzr;

rlpmr = dr(jmr:lndz); for i=1:rln rlpmrr(i)=dr(jmr-i+1)-min(rlpmr);

```



```

end rlpmrr = abs(rlpmrr);

radiip = sqrt(rlpmrr .* rlpmrr + rlpmzr .* rlpmzr); zrg= rlpmrr ./
rlpmzr ;
mzrg = radiip ./ zrg ;%./ radiip ;
mzrg2= mzrg ./ zrg ; iimr = find(mzrg2 == max(mzrg2(2:rln)));
jimr=jmr+iimr-1;

%*****
lnbkg = lndz-floor(0.7 .* rln); bkgr = mean(dz(lnbkg:lndz)); bkgrmin
= min(dz(lnbkg:lndz));

difbkgr = abs(dz(jmr:lndz) - bkgr); [bj]=find(difbkgr ==
min(difbkgr)); if length(bj) >1 bj = bj(1,1); end bj=bj;
jnpr= min(bj)+jmr-1; % bkg -- mean of the background

[iipr]= find(dz(jmr:lndz) == bkgrmin ); jipr = iipr + jmr -1;

hold on plot(dr(jnpr),dz(jnpr),'*m')
plot(rlpr,bkgr*ones(size(rlpz)),'-m') plot(dr(jimr),dz(jimr),'*c')
plot(rlpr,bkgrmin*ones(size(rlpz)),'-r')

% NB conditions have to be set in order to choose which of the above
% three methods should be used as bkg
% find the point between max(pile up) and min(apex) == bkg
% to calculate the diagonal excluding the pile up
% select the background to use
% in most cases mean should be used
% TRY AND INTRODUCE SOME TERM TO EVALUATE THE WHICH BKG TO USE

difbkr = abs(dz(jn:jtr) - bkgr); [i,j] = find(difbkr == min(difbkr))
; jepr = j +jn -1 ;

```

```

if length(jepr) >1 eppt =abs(jtl-jepr); ep=find(eppt == min(eppt));
jepr = jepr(ep); else jepr = jepr end

% plots the point excluding the pile up
hold on plot(dr(jepr),dz(jepr),'*g')

if length(jtr) <1 drtr = 'err' drtz = 'err' drtr = 0 ; drtz = 0 ;
else
drtr = dr(jtr);      % turning of indent
drtz = dz(jtr);      % turning pt of indent
end

if length(jmr) <1 drmr = 'err' dr mz = 'err' drmr = 0 ; dr mz = 0 ;
else
drmr = dr(jmr);      % max of indent
dr mz = dz(jmr);      % max of indent
end

if length(jnpr) <1 dripur = 'err' dripuz = 'err' dripur = 0 ; dripuz
= 0 ; else
dripur = dr(jnpr);      % includes pile up
dripuz = dz(jnpr);      % includes pile up
end

if length(jimr) <1 drimpur = 'err' drimpuz = 'err' drimpur = 0 ;
drimpuz = 0 ; else
drimpur = dr(jimr);      % includes pile up true value
drimpuz = dz(jimr);      % includes pile up true value
end

if length(jepr) <1 drepur = 'err' drepuz = 'err' drepur = 0 ; drepuz
= 0 ; else

```

```

drepur = dr(jepr); % excludes pile up
drepuz = dz(jepr); % excludes pile up
end

if length(jn) <1 drmin = 'err' dzmin = 'err' drmin = 0 ; dzmin = 0 ;
else drmin = dr(in,jn); dzmin = dz(in,jn) ; end

disp('ptsrgh = drtr drmr dripur drimpur drepur ');

ptsright = [drtr drmr dripur drimpur drepur drmin;
            drtz drnz dripuz drimpuz drepuz dzmin] ;

% THIS CONCLUDES THE ROUTINE FOR IDENTIFYING 3 POINTS TO THE LEFT
%      OF THE APEX
% *****

VolArea

function [volume,volumepu] =
volarea(B,diaglen,XX1,YY1,XX2,YY2,scansiz,truez)

B=B; diaglen=diaglen; XX1=XX1; YY1=YY1; XX2=XX2; YY2=YY2;
scansiz=scansiz;

dig1 = [diaglen(1,:) diaglen(3,:)]; dig2 = [diaglen(6,:)
diaglen(8,:)];
dig1r = [diaglen(3,:) diaglen(6,:)]; % point(index) in dR1
dig2r = [diaglen(10,:) diaglen(13,:)]; %point(index) in dR2

digxx1=XX1(dig1r); digyy1=round(YY1(dig1r));

digxx2=round(XX2(dig2r)); digyy2=YY2(dig2r);

%Turning point

```

```

tpl1 = [digxx1(3) digyy1(3)]; tpr1 = [digxx1(8) digyy1(8)]; tpl2 =
[digxx2(2) digyy2(2)]; tpr2 = [digxx2(8) digyy2(8)];
%figure,
subplot(2,2,1) plot(XX1,YY1,'.g',XX2,YY2,'.m'),zoom
    hold on, plot(tpl1(1),tpl1(2),'*r')
    hold on, plot(tpr1(1),tpr1(2),'*m')
    hold on, plot(tpr2(1),tpr2(2),'*g')
    hold on, plot(tpl2(1),tpl2(2),'*w')
pts_tp= [tpl1; tpl2; tpr1 ;tpr2];
    [vol_tp,voltp,nntp,linmc,pts_tp] = volpts(B,pts_tp) ;

% excluding Pile up == background

epul1 = [digxx1(4) digyy1(4)];
epur1 = [digxx1(7) digyy1(7)];
epul2= [digxx2(4) digyy2(4)];
epur2 = [digxx2(7) digyy2(7)];
%figure,
subplot(2,2,2) plot(XX1,YY1,'.g',XX2,YY2,'.m'),zoom
    hold on, plot(epul1(1),epul1(2),'*r')
    hold on, plot(epur1(1),epur1(2),'*m')
    hold on, plot(epur2(1),epur2(2),'*g')
    hold on, plot(epul2(1),epul2(2),'*w')
pts_epu= [epul1; epul2; epur1 ;epur2];
    [vol_epu,volepu,nnepu,linmc,pts_epu] = volpts(B,pts_epu) ;

% Maxima

ml1 = [digxx1(2) digyy1(2)];
mr1 = [digxx1(9) digyy1(9)];
ml2 = [digxx2(2) digyy2(2)];
mr2 = [digxx2(9) digyy2(9)];

```

```

subplot(2,2,3) plot(XX1,YY1,'.g',XX2,YY2,'.m')
    hold on, plot(ml1(1),ml1(2),'*r')
    hold on, plot(mr1(1),mr1(2),'*m')
    hold on, plot(mr2(1),mr2(2),'*g')
    hold on, plot(ml2(1),ml2(2),'*w')
    pts_max= [ml1; ml2; mr1 ;mr2];
    [vol_max,volmax,nnmax,linmc,pts_max] = volpts(B,pts_max);

% Including pile up

ipul1 = [digxx1(1) digyy1(1)]; ipur1 = [digxx1(10) digyy1(10)];
ipul2 = [digxx2(1) digyy2(1)]; ipur2 = [digxx2(10) digyy2(10)];
%figure,
subplot(2,2,4) plot(XX1,YY1,'.g',XX2,YY2,'.m'),zoom
    hold on, plot(ipul1(1),ipul1(2),'*r')
    hold on, plot(ipur1(1),ipur1(2),'*m')
    hold on, plot(ipur2(1),ipur2(2),'*g')
    hold on, plot(ipul2(1),ipul2(2),'*w')
pts_ipu= [ipul1; ipul2; ipur1 ;ipur2];

[vol_ipu,volipu,nnipu,linmc,pts_ipu] = volpts(B,pts_ipu);

[gvol,Tvolipu,svol,nntrueipu,tzbkg] =
    volrad(B,pts_ipu,pts_tp,vol_tp,truez);

volumeipu=[sum(sum(gvol)), sum(sum(Tvolipu)), sum(sum(svol)),
nntrueipu, tzbkg];

disp('IPU, Max, TP, EPU')
```

```

volume=[sum(sum(volipu)),sum(sum(volmax)),sum(sum(voltp)),sum(sum(volepu));
        sum(sum(vol_ipu)),sum(sum(vol_max)),sum(sum(vol_tp)),sum(sum(vol_epu));
        nnipu nnmax nntp nnepu] ;

```

Volpts

```

function [gvol,voltrue,nnpoints,linmc,ptsvol] = volpts(B,ptsvol)

```

```

B = B;

```

```

%pts = getpts

```

```

pts=ptsvol;
ptsA = pts(1,:);
ptsB = pts(2,:);
ptsC = pts(3,:);
ptsD = pts(4,:);

```

```

d1pts = [ptsA ; ptsB];
d2pts = [ptsB ; ptsC];
d3pts = [ptsC ; ptsD];
d4pts = [ptsD; ptsA];

```

```

V1 = d1pts; V2 = d2pts; V3 = d3pts; V4 = d4pts;

```

```

d1=polyfit(V1(:,1),V1(:,2),1);
d2=polyfit(V2(:,1),V2(:,2),1);
d3=polyfit(V3(:,1),V3(:,2),1);
d4=polyfit(V4(:,1),V4(:,2),1);

```

```

linmc =[d1;d2;d3;d4];

```

```

gg = (1:256)' * ones(size(1:256));

ab = gg - d1(1)*gg';
bc = gg - d2(1)*gg';
cd = gg - d3(1)*gg';
da = gg - d4(1)*gg';
%Bmax=max(max(B))-B ;
gvol = zeros(size(B)) ;
voltrue = zeros(size(B));
[i,j] = find(ab > d1(2) & bc > d2(2) & cd < d3(2) & da < d4(2));
if i ~=[] & j ~=[] V=[i,j] ;
[a,b] = size(V) ;
for n =1:a
gvol(V(n,1),V(n,2)) = B(V(n,1),V(n,2)) ;
%voltrue(V(n,1),V(n,2)) =Bmax(V(n,1),V(n,2)) ;
end end hold on, spy(gvol,'c')

```

```

S=nonzeros(gvol);
volt=max(S)-S;
voltrue = sum(volt);
nnpoints = nnz(gvol);

```

Volrad

```

function [gvol,Tvolipu,svol,nntrueipu,tzbkg] =
volrad(B,pts_ipu,pts_tp,vol_tp,truez)

B = B;
%pts = getpts
pts=pts_ipu; ptssub=pts_tp; volsub =vol_tp; truez=truez;

ptsA = pts(1,:);
ptsB = pts(2,:);

```

```

ptsC = pts(3,:);
ptsD = pts(4,:);

d1pts = [ptsA ; ptsB];
d2pts = [ptsB ; ptsC];
d3pts = [ptsC ; ptsD];
d4pts = [ptsD; ptsA];

V1 = d1pts;
V2 = d2pts;
V3 = d3pts;
V4 = d4pts;

d1=polyfit(V1(:,1),V1(:,2),1);
d2=polyfit(V2(:,1),V2(:,2),1);
d3=polyfit(V3(:,1),V3(:,2),1);
d4=polyfit(V4(:,1),V4(:,2),1);

linmc =[d1;d2;d3;d4];

gg = (1:256)' * ones(size(1:256));

ab = gg - d1(1)*gg';
bc = gg - d2(1)*gg' ;
cd = gg - d3(1)*gg' ;
da = gg - d4(1)*gg' ;
gvol = zeros(size(B)) ;
voltrue = zeros(size(B));

minB = min(min(B));
[oxB,oyB] = find(B == minB) ;
oxB = oxB(1);
oyB = oyB(1);

```



```

    OB = [oxB,oyB,B(oxB,oyB)] ;
    Omtx = ones(size(B)) ;
    for
i=1:length(B) ;
    dfx = (i-oyB) * Omtx(:,i) ;
    distx(:,i) = abs(dfx) ;
end ;

for j=1:length(B);
    dfy = (j-oxB) * Omtx(j,:);
    disty(j,:) = abs(dfy);
end ;
    dist = sqrt(disty .* disty + distx .* distx ) ;

y=pts(:,2)-OB(1); x=pts(:,1)-OB(2); r=sqrt(x .* x + y .* y);

rr=mean(r);
ptrad=rr * 1.20;
rad=zeros(size(B));
rad=(dist <=ptrad);

[i,j] = find(rad ~= 0);
    if i ~=[] & j ~=[]
        V = [i,j] ;
        [a,b] = size(V) ;
        for n =1:a
            gvol(V(n,1),V(n,2)) =B(V(n,1),V(n,2)) ;
        end
    end
end
figure,spy(gvol,'g'),hold on,spy(vol_tp),zoom
hold on,contour(B,32)
figure,imagesc(gvol-vol_tp),colorbar,colormap('jet')
```

```

nnipu = nnz(gvol); S=nonzeros(gvol);

Ssub=nonzeros(vol_tp); nntp=nnz(vol_tp);

% background subtraction

tz=[truez(:,1) truez(:,3:4)];

tzbkg=mean(mean(tz));
nntrueipu= nnipu-nntp;
Tvolipu = gvol - vol_tp;
sipu=nonzeros(Tvolipu);
smin=min(min(sipu));
svol=zeros(size(Tvolipu));
[i,j] = find(Tvolipu > 0);
if i ~=[] & j ~=[] V =[i,j] ;
    [a,b] = size(V) ;
    for n =1:a
        svol(V(n,1),V(n,2)) =Tvolipu(V(n,1),V(n,2)) -smin;
    end
end

LineAng

function
[trueangpt,truedz]=lineang(B,diaglen,truediag,truez,scansiz,zcal)

B=B; di=diaglen; truediag=truediag; truez=truez; scansiz=scansiz;
zcal=zcal;

angval = truez ./ (truediag/2); angdiagz = 90 - atan(angval)*180/pi;
truedz=mean(angdiagz);

```

```

d1l=abs([di(1,2)-di(1,5),di(1,3)-di(1,5),di(1,1)-di(1,2),di(1,2)-di(1,3)]);
z1l=abs([di(2,2)-di(2,5),di(2,3)-di(2,5),di(2,1)-di(2,2),di(2,2)-di(2,3)]);
d1r=abs([di(4,1)-di(4,4),di(4,1)-di(4,3),di(4,5)-di(4,4),di(4,4)-di(4,3)]);
z1r=abs([di(5,1)-di(5,4),di(5,1)-di(5,3),di(5,5)-di(5,4),di(5,4)-di(5,3)]);

d2l=abs([di(1,2)-di(1,5),di(1,3)-di(1,5),di(1,1)-di(1,2),di(1,2)-di(1,3)]);
z2l=abs([di(2,2)-di(2,5),di(2,3)-di(2,5),di(2,1)-di(2,2),di(2,2)-di(2,3)]);
d2r=abs([di(4,1)-di(4,4),di(4,1)-di(4,3),di(4,5)-di(4,4),di(4,4)-di(4,3)]);
z2r=abs([di(5,1)-di(5,4),di(5,1)-di(5,3),di(5,5)-di(5,4),di(5,4)-di(5,3)]);

angtmp=90-atan([(z1l ./ (d1l)) (z1r ./ (d1r));
                (z2l ./ (d2l)) (z2r ./ (d2r));])*180/pi

disp('max-min,tp-min,ipul-max,max-tp,max-min,tp-min,inpul-max,max-tp')
trueangpt=mean(angtmp);

```

Appendix E

Elastic recovery of the indentation

Recovery of indentation has been discussed in the macro-scale regime by several authors eg. Tabor [Tabor, 1951], Samuels [Samuels and Mulhearn, 1957], Mulhearn [Mulhearn, 1959], Sneddon [Sneddon, 1946], Dugdale [Dugdale, 1953], Timoshenko [Timoshenko and Goodier, 1951], Hirst [Hirst and Howse, 1969], Johnson [Johnson, 1970], Bishop [Bishop et al., 1945], [Perrott, 1977], [Pethica et al., 1983], [Samuels, 1986], [Chiang et al., 1982] and the study of recovery during indentation on a sub-micron scale has become possible with nanoindentation which allows for continuous monitoring of the force (load) and depth displacement of the indenter [Oliver et al., 1986],[Pharr and Cook, 1990], etc. In microindentations the recovery profiling is possible after unloading, by comparing the geometry of the Vickers diamond indenter with that of the three-dimensional AFM measured residual indentation.

A residual indent is a signature of the resistance to deformation of a specimen under stress during indentation. The morphology of the residual indents is given by the AFM images and one can compare the shape and size of residual indent with that of the Vickers diamond indenter geometry. The recovery of indentation for WC-Co samples can be determined by either three-dimensional comparisons or linear comparisons e.g. of diagonal, depth or angle. Figure 5.6 (page 55) shows the anisotropic recovery of the material when unloaded, during the relaxation process the “behaviour” is mostly elastic. It is elastic plastic during loading.

The recovery is manifested by change in the diagonals length, depth, angle and volume of the residual indent relative to the diamond Vickers indenter. Although, since the cemented carbides have mechanical properties (hardness, Young’s mod-

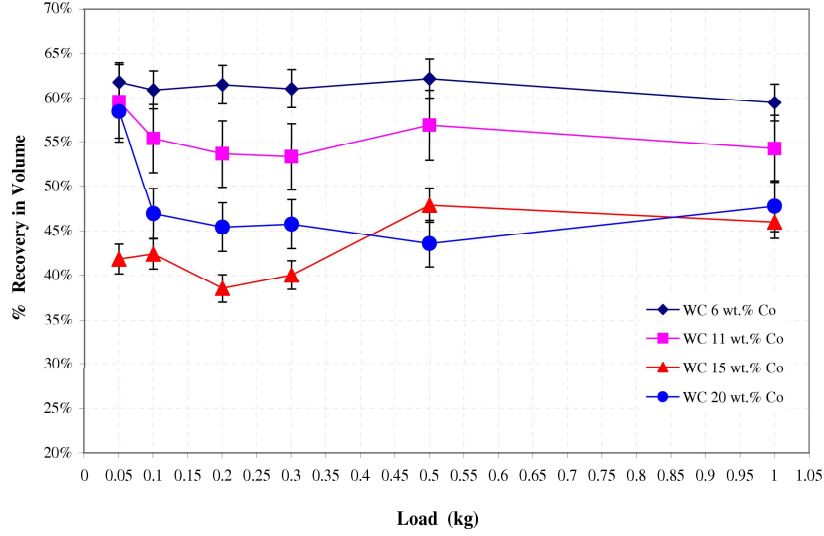


Figure E.1: Recovery in the volume of the indentation between loading and unloading during indentation.

ulus, Poisson ratio) which are high even when compared to diamond, the Vickers diamond indenter experiences elastic strains during indentation. The elastic strain experienced by the Vickers diamond indenter at each indenting load for each given cement carbide were determined by finite element analysis (see appendix A).

E.0.1 Recovery in indentation volume

The volume V_{Th} of the indenter under load is $V_{Th} = d_{Th}^2 h_{Th} / 6 = d_{Th}^3 / 42$, where $d_{Th} = \sqrt{1.854 \times L / H_0}$ is the diagonal of the indent under load, assuming a constant hardness H_0 of the sample. In figure E.1 the percentage recovery of the volume is given as $(V_{Th} - V_{RI}) / V_{Th}$, where V_{RI} is the volume of the residual indent. The recovery in volume for the WC-Co samples is between 40% and 60%. For the WC 6 wt.% Co the recovery is constant for all indenting loads tested at $\approx 62\%$, while for the for WC 11wt.% Co and WC 20 wt.% Co there is an average recovery of 55% and 47%.

E.0.2 Recovery of indentations diagonal, depth and angle

The residual indent can be compared with the Vickers diamond indenters geometry to determine its recovery, taking into account the elastically deformed diamond indenter under load. Since its difficult to measure the diagonal of indentation formed under load, assuming that the hardness H_0 (= macrohardness) is a constant for all loads, from the hardness equation we can determine the diagonal under load to be

$$d_{Th} = \sqrt{2 \sin 68 \frac{L}{H_0}} - d_e$$

where d_e is due to the elastic deformation of the diamond indenter as determined by FEA. The corresponding depth of indentation is

$$h_{Th} \simeq d_{Th} / (2 \tan 74^\circ) \simeq d_{Th} / 7.$$

Due to the change in the diagonal and depth (tip deflection) the angle between the opposite the edges of the diamond indenter changes as well.

Figure E.2 is a sketch of the line profile along opposite edges of residual indentation and the Vickers diamond indenter. These line profiles indicate recovery of the indentation on unloading and formation of the “pile-ups” on unloading. The line profile marked “actual line profile” is a line profile along the opposite edges of an AFM measured residual indentation, with the piled-up material around the residual indent exaggerated. The solid line marked “Th” represents the line profile of the indenter under loading with diagonals d_{Th} and depth h_{Th} .

Also shown in the figure are four additional indenter line profile corresponding to the measured diagonals d_I , d_T , d_M (for notations see appendix B) and measured depth h_m and its corresponding “theoretical” diagonal d_{h_m} based on Vickers diamond indenter geometry. A consequence of using the depth h_m for calculating the hardness of a material is that it underestimates the contact area which influences the calculated hardness. While h_{pu} is the height of the pile-ups. The semi-circular dotted lines represents the hemispherical region of the material affected by the indentation under the indenter/surface.

The recovery in the diagonal is evaluated between the diagonals d_{Th} and d_I and is given in figure E.3. Similarly, the recovery in depth is evaluated between the depth h_{Th} and the measured depth h_m as shown in figure E.4.

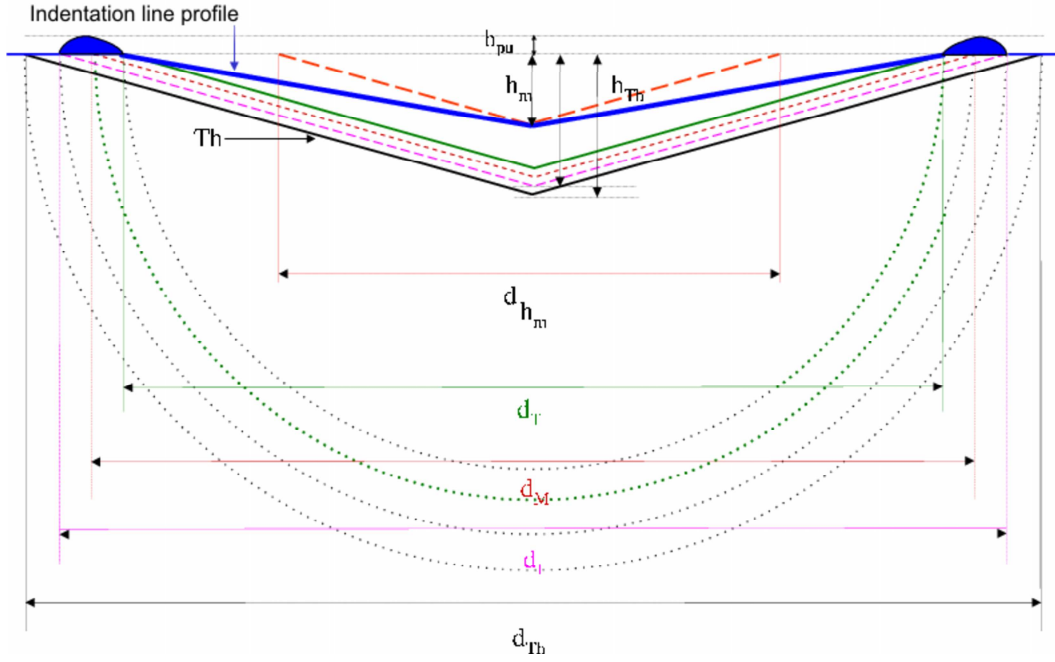


Figure E.2: Sketch of the line profile measured and expected (if no relaxation occurred) based on indenter geometry. The labels are discussed in text. [Note: In the sketch the ratio of $d_{Th}/h_{Th} = 7$ to scale, and the angle subtended between the "V" lines is 148.1° .]

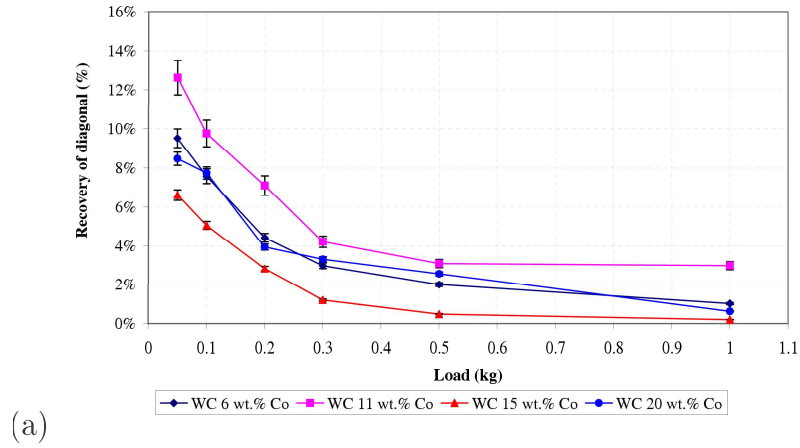


Figure E.3: Recovery of the diagonal due to elastic relaxation.

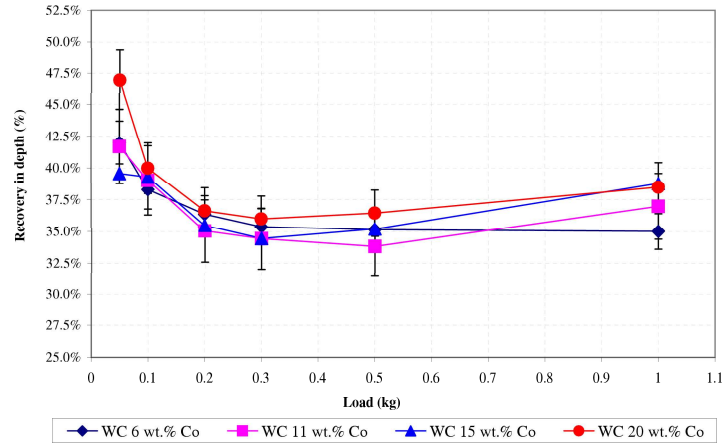


Figure E.4: Recovery of the indentation depth.

The elastic recovery of the diagonal for Vickers indenter has been established to be 7-8% [Stilwell and Tabor, 1962], [Samuels and Mulhearn, 1957]. From the results of the diagonals (figure 5.3) and the recovery in diagonal (figure E.3) the pile-ups of the material are expected to form during relaxation process and as shown in figure E.5, their shape is affected by the volume of the binder cobalt content. A change in the shape of the pileups is observed between WC 11 wt.% Co and WC 15 wt.% Co as shown schematically in figure E.5.

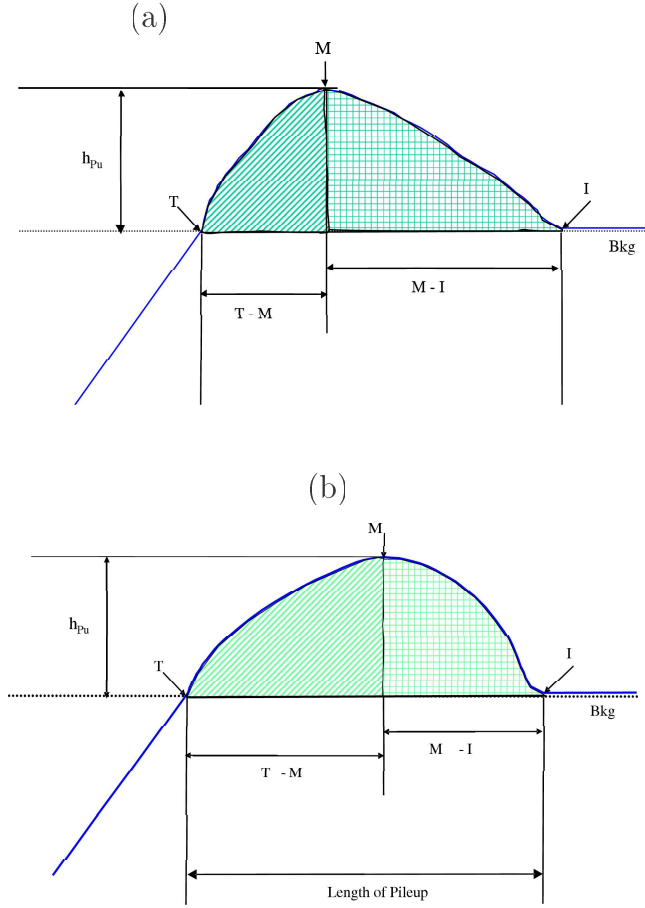


Figure E.5: Schematic of the shift in the shape of pile-ups of the material formed during unloading between cobalt binder content of ≤ 11 wt.% (a) and ≥ 15 wt.% (b) for the tested samples. The notations T , M and I are explained in figure 5.1 and h_{pu} is the height of the pileups.

Appendix F

An empirical Correction factor for ISE

The results of the tests on WC-Co which were reported in chapter 5 are shown again in figure F.1 (dotted lines) for the four WC-Co grades having mean WC grain size $1 - 1.2\mu\text{m}$ and Co content ranging from 6 to 20 wt%. The results joined by dotted lines in figure F.1 were obtained from the length of the diagonals (d_I) of the hardness indentations (after removal of the indenting load) by applying the formula

$$H_m = 2 \sin \alpha \frac{L}{d_m^2} \quad (\text{F.1})$$

where: H_m is the measured microhardness, P the applied load, $\alpha = 68^\circ$ is the half-angle between opposite triangular faces of the pyramidal Vickers diamond indenter and d_m the measured mean diagonal of the indentation, and $d_m^2/(2 \sin \alpha)$ is the contact area between indenter and indented material.

In the figure F.1 a second set of results joined by full lines which were obtained by applying the following formula

$$H_0 = \eta \ 2 \sin \alpha \frac{P}{d_m^2} \quad (\text{F.2})$$

where η is a correction factor which was found empirically figure F.2 to be

$$\eta = \exp - \left(\frac{A_0 - A_m}{A_0} \right) \quad (\text{F.3})$$

where H_0 is the macrohardness: (i.e. the hardness measured at 1kg indenting load), A_0 contact area between indenter and indented material under load P and A_m is the contact area between indenter and indented material after unloading.

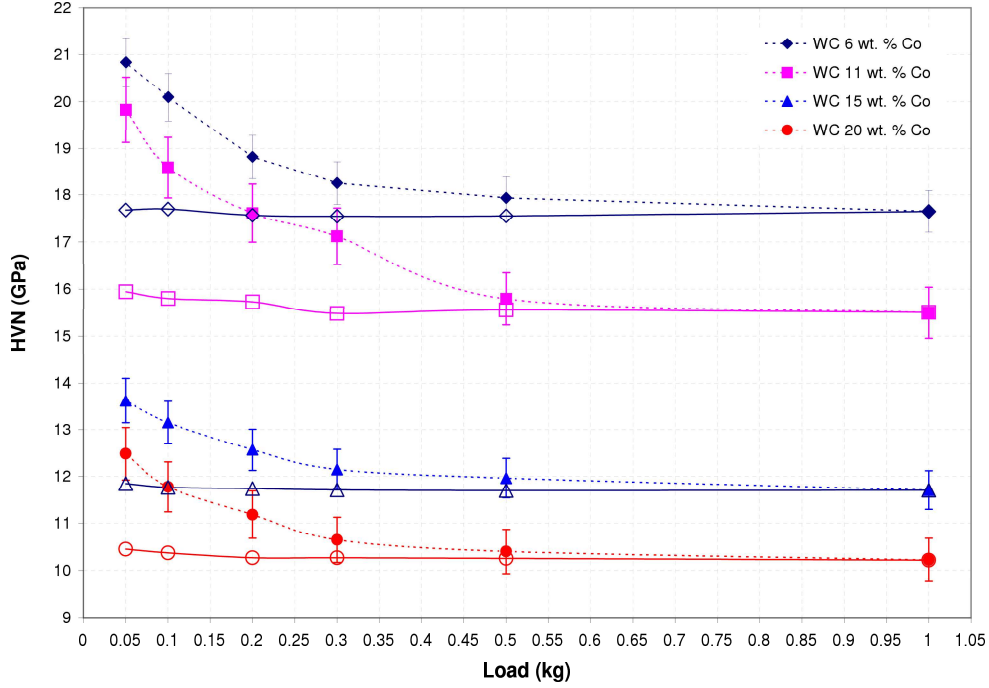


Figure F.1: Hardness (solid symbols) and hardness obtained by applying the correction factor (open symbols) for the four carbide grades versus applied load.

A_m was measured by (AFM) and A_0 was calculated assuming that the hardness of the material should be constant at all loads and taking the macrohardness as the assumed constant hardness. By applying the definition of hardness one has

$$H_0 = \frac{P}{A_0} \quad (\text{F.4})$$

With increasing load P , A_0 increases. In equation (F.2) and in the second set of results in figure F.1 the angle α was assumed to be 68° . However, it has been found by AFM that α increases with decreasing load and with decreasing contact area after removal of the applied load. The maximum value of α was found by AFM to be 76° in WC single crystals (when measuring the hardness of the (0001) plane) [Luyckx et al., 1999] at an indenting load of 25 g.

The exponential correction factor is consistent with the following considerations based on the definition of hardness (equation F.4). If H_0 is the macrohardness of the material and H_m is the measured hardness at a load P and we assume that

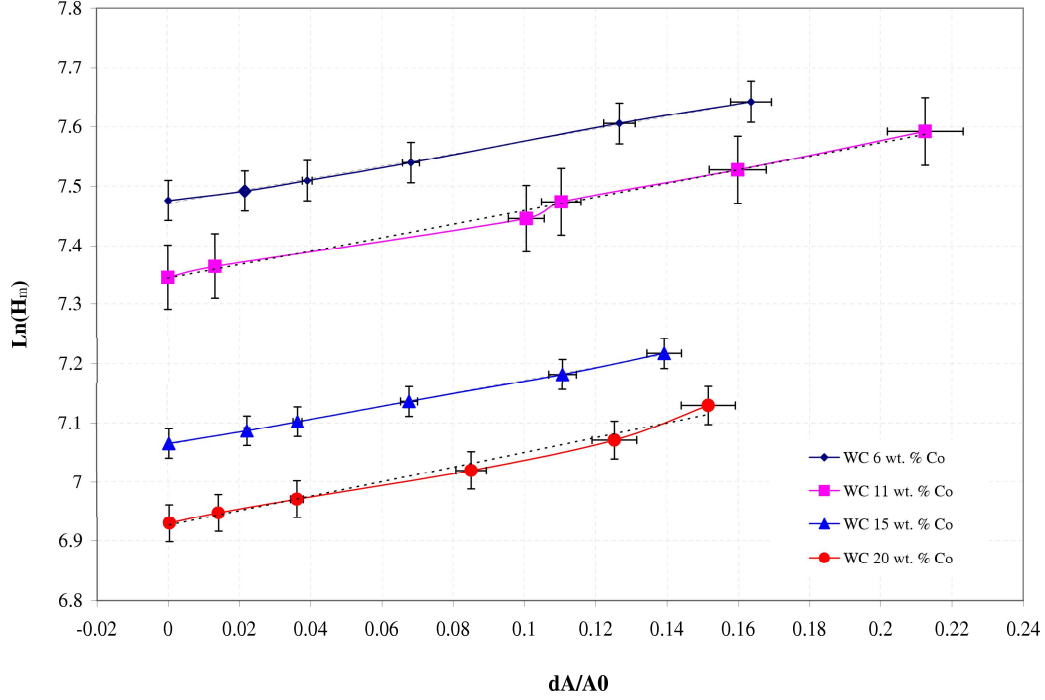


Figure F.2: Logarithm of Hardness versus the ratio of the difference between contact areas ($A_0 - A_m$) to the contact area under load (A_0) for loads ranging from 50g to 1kg. Where A_m is the size of the contact area after unloading.

the "real" microhardness of the material is constant ($= H_0$) but due to elastic relaxation the measured hardness is H_m for the same applied load P

$$H_0 = \frac{P}{A_0} \quad \text{and} \quad H_m = \frac{P}{A_m} \quad (\text{F.5})$$

The difference in hardness $H_0 - H_m$ is $H_0 - H_m = P(1/A_0 - 1/A_m)$ or H_0 and H_m for the same load P are

$$H_0 = H_m \frac{A_m}{A_0} \quad (\text{F.6})$$

If $(A_0 - A_m)/A_0 \ll 1$ the exponential factor (equation F.3) can be written in the form

$$\exp - \left(\frac{A_0 - A_m}{A_0} \right) \simeq 1 + \left(- \frac{A_0 - A_m}{A_0} \right) + \dots \simeq \frac{A_m}{A_0} + \dots \quad (\text{F.7})$$

where the dots represent terms of second or higher order of $1 - A_m/A_0$.

Combining equation F.2 F.3 and F.6 gives the equation for the correction factor for the hardness as

$$H_0 \simeq H_m \exp - \left(\frac{A_0 - A_m}{A_0} \right) \quad (\text{F.8})$$

As a first order approximation equation F.8 reduces to equation F.6.

It must be noted that the factor in equation F.8 has been found to “eliminate” the ISE in most materials for which ISE has been reported in the literature [Gong et al., 1998a], [Gong et al., 1998b], [Stelmashenko et al., 1993], [Li and Brandt, 1992], also for Knoop hardness tests [Hays and Kendall, 1973], examples are Vickers and Knoop indentation are given in figure F.3.

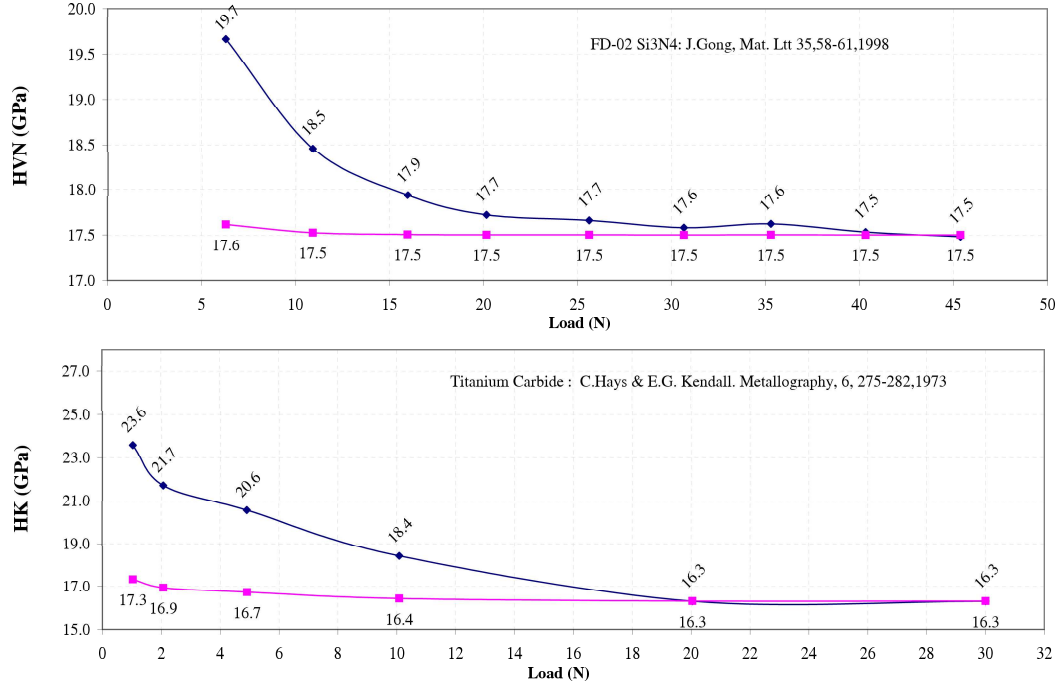


Figure F.3: The correction factor is applied to data from the literature ([Gong et al., 1998b], [Hays and Kendall, 1973]) for Vickers indenter and Knoop indenter respectively.

Appendix G

Plasticity characteristics of hardness measurements

“Plasticity characteristic” of hardness was introduced by Milman “[Milman et al., 1993]” to explain indentation size effect. There is a good correlation between the present experimental measurements on WC-single crystal and the theory proposed by Milman *et al.* [Milman et al., 1993], [Galanov et al., 1999]. In this section a letter from Milman is reported, which was received after the attached paper in Journal of Material Science Letters [Luyckx et al., 1999] (see appendix H).

APPENDIX G. PLASTICITY CHARACTERISTICS OF HARDNESS MEASUREMENTS

I congratulate you with the very important results published in S.Luyckx et al. «The effect of elastic relaxation...» J.of Mater. Sci.Letters, v.18 (1999) p.705-706.

In this work you have measured angles of the indentation pyramid on WC at different loads.

I dreamed about these results many years to control our theories.

In our theory (I am sending you this paper «Investigation of high-hard materials mechanical properties by indentation» B.A. Galanov, Yu.V. Milman et al.) the angle between pyramid faces γ_2 ($\gamma_2 = \alpha$ in your paper) can be calculated from

$$\text{ctg}\gamma_2 = \text{ctg}\gamma_1 - 1.77 \frac{HM}{E_{ef}} \quad (1)$$

Here $\gamma_1 = 68^\circ$; HM is the Meier hardness

$$HM = \frac{HV}{\sin 68^\circ};$$

$$\frac{1}{E_{ef}} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2}$$

In the first approximation I took for diamond $\nu_1 = 0.1$; $E_1 = 1200$ GPa and for WC $\nu_2 = 0.31$ and $E_2 = 700$ GPa. In this case $E_{ef} = 473$ GPa.

In the table 1 you see the comparison of your experimental and calculated values of γ_2 .

The average plastic deformation on the contact area indenter-specimen ϵ_p can be calculated from (see the same paper)

$$\epsilon_p = \ln \sin \gamma_2 \quad (2)$$

In the table you see both values of ϵ_p , calculated from your experimental values of γ_2 by Eq.2 ($\epsilon_p(\text{exp})$) and using γ_2 calculated from Eq.(1) ($\epsilon_p(\text{calcul})$).

Average elastic deformation can be calculated from (see the same paper).

$$\epsilon_e = -1.08 \cdot \left(1 - \nu_2 - 2\nu_2^2\right) \frac{HV}{E} \quad (3)$$

(Here (-) is because we have compression deformation).

And the total deformation

$$\epsilon_t = \epsilon_p + \epsilon_e \quad (4)$$

ϵ_e and ϵ_t are given in the table 1 too.

$$\epsilon_{t(\text{exp})} = \epsilon_{p(\text{exp})} + \epsilon_e \quad \text{and} \quad \epsilon_{t(\text{calcul})} = \epsilon_{p(\text{calcul})} + \epsilon_e$$

Table 1.

P, g	HV, GPa	γ_2 (exp)	γ_2 (calcul)	ϵ_p , % (exp)	ϵ_p , % (calcul)	ϵ_e , %	ϵ_t , % (exp)	ϵ_t , % (calcul)
25	34.08	76.0	75.0	3.02	3.43	3.88	6.90	7.31
50	30.29	74.8	74.3	3.56	3.82	3.45	7.01	7.27
100	30.20	71.7	74.3	5.19	3.82	3.44	8.63	7.26
200	23.72	70.6	72.9	5.84	4.54	2.70	8.54	7.24
500	20.43	71.1	72.2	5.54	4.92	2.33	7.87	7.25
our results								
50	30.60		74.3		3.78	3.49		7.27
100	27.16		73.6		4.15	3.10		7.25
200	20.59		72.2		4.90	2.35		7.25
500	16.70		71.4		5.36	1.90		7.26
1000	15.17		71.1		5.54	1.79		7.33
5000	11.00		70.2		6.10	1.25		7.35

Figure 1

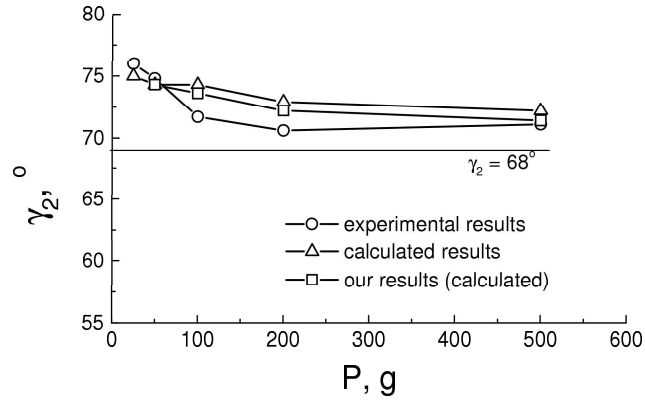
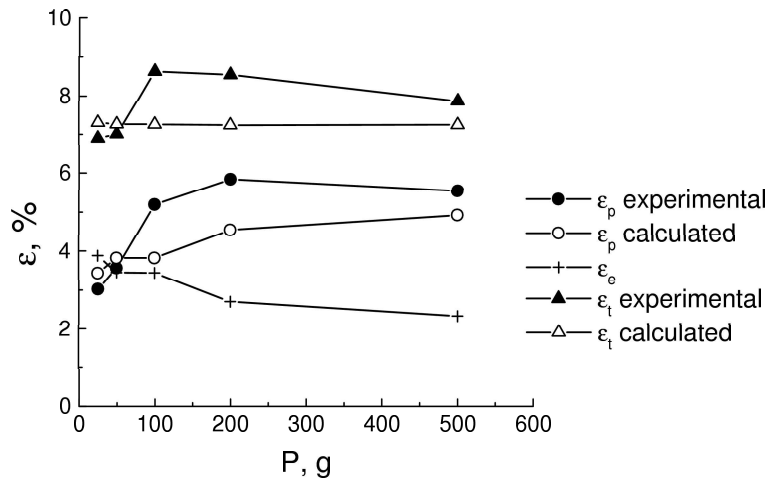


Figure 2



Now discussion.

1. The correlation of the experimental and calculated values of γ_2 is very good for the low loads (20 g and 50 g) and not so good for the bigger loads (see the fig.1). But in common this correlation is good.

We have measured hardness on your WC single crystals before at loads 0.05 kg; 0.1 kg; 0.2 kg; 0.5 kg; 1 kg and 5 kg. The values of HV are given in the table 1.

You see that our values of HV for 0.1 kg; 0.2 kg and 0.5 kg are smaller then your.

In the table 1 I calculated values of γ_2 ; ϵ_e , ϵ_p and ϵ_i for our results too. In this case correlation with your experimental values of γ_2 is better (see the table 1 and fig.1).

By this way I think your results confirm our theory and Eq.(1).

2. Now you have experimental dependence of ϵ_e , ϵ_p and ϵ_t on the indentation load (table 1 and fig.2). And you see that ϵ_p decreases if P decreases, but ϵ_e increases.

In my work (Yu.V.Milman. The dependence of hardness on the load on indenter and hardness at the fixed indentation diagonal. Problems of Strength, v.6, 1990, p.52-56 [In Russian]) I have supposed that if the diagonal of indentation d decreases, the plastic deformation must be more difficult and ϵ_p must be lower. The total deformation during indentation is approximately constant and for this reason ϵ_e must increase if P (and d) decreases. If ϵ_e increases the hardness must increase too from the Gook low of the type (3). By this way I have explained the scale dependence of hardness - hardness increases with decreasing P and d. Now you have given the confirmation of this concept. I think we could discuss these problems in the joint paper.

Communication date: January 13, 2000.

Appendix H

Papers

Papers published during the course of this work are attached here.

1. “Automated analysis of Vickers Microindentations by Atomic Force Microscopy”, Sanjiv Shrivastava and S Luyckx, Proceedings, AES - ATEMA’ 2007 International Conference, Montreal, Canada, August 06 -10, 2007, pp. 27 - 34.
2. “The size effect in microindentation”, Nabarro, F. R. N. and Shrivastava, S. and Luyckx, S. B.,Philosophical Magazine Vol.86, pg.4173-4180, 2006.
3. The effect of elastic relaxation on the indentation size effect in tungsten carbide, Luyckx, S., Demanet, C.M. and Shrivastava, S., Journal of Materials Science Letters, Vol 18, pg 705-706, (1999)

*S. Shrivastava and S. Luyckx / Proceedings, AES – ATEMA' 2007 International Conference,
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Automated analysis of Vickers's Microindentations by Atomic Force Microscopy

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Abstract

Atomic Force Microscopy (AFM) images of Vickers's microindentations for four different grades of WC-Co cemented carbides with varying cobalt content and constant tungsten-carbide grain size were analyzed using the program AutoIndent written specifically for the project

Keywords

Microindentation, Hardness, AFM, WC-Co, pileups,

1 Introduction

The Vickers hardness of a material is determined from the diagonals of the residual indent. It is common practice to measure the diagonals of Vickers indentations by optical microscopy (OM) but the disadvantage of OM

measurements for microindentations in hard materials such as WC-Co is that the size of the indentations at the lowest loads is of the order of the resolution limits of OM. Other possible techniques to measure indentations, such as differential interference contrast microscopy (DIC), and scanning electron microscopy have similar limitations which are due to instruments resolutions and operator-based subjective error. An alternative method of determining the hardness is by using continuous depth-sensing indentation techniques.

Atomic force microscopy (AFM) is amongst the more recent techniques employed in residual indentation measurements. AFM provides true three-dimensional information on the indent and its surroundings with resolution of the order of sub-nanometres in lateral and vertical direction. This paper discusses the advantages of the automated software

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AutoIndent specifically written for the analysis of the AFM image of Vickers microindentation on WC-Co cemented carbides. The paper deals with the description of the method developed, the parameters determined in characterizing the residual indents and problems encountered with commercial AFM packages or indentation analysis software (ProScan[1], SPIP[2], NanoMc[3], WSxM[4], Nanoscope III [5], etc.). The method used in AutoIndent has solved problems previously encountered in both line and surface measurements in the estimation of diagonals, area and volume of indentations.

Indentation analysis by commercial software programs (SPIP, NanoMc, etc.) determines parameters such as surface area, projected area and the volume of residual indent by defining the region of interest (ROI). In the case of indentation analysis there are two features of interests (i) the residual indent and (ii) the piled-up material. The ROI is estimated (or defined) by using: (i) Threshold method – commonly used by most AFM analysis software (eg, ProScan, WSxM, Nanoscope III) (ii) Height tangent method - (NanoMc) (iii) Histogram analysis or pore analysis (SPIP) methods. In addition, other parameters measured are the contact depth and the residual indent angle. Shuman [6] has compared the indentation on AISI 1020 steel sample by using NanoMc, Nanoscope III and SPIP softwares. A good correlation between NanoMc and SPIP is observed for most of the parameters measured, except for those parameters related to the piled-up material. While in case of Nanoscope III, most of the parameters measured are underestimated in comparison to the NanoMc and SPIP measurements.

The three methods used for identifying the residual indent and piled-up material are subject to system error or operator's choice error. The height tangent method provides a systematic way of selecting the ROI for the residual indent. In this method a threshold value of 90% of the maximum height within the AFM image (SPIP and NanoMac) is taken. Firstly, there is no justification for using 90% of maximum height and secondly, the ROI thus selected for the residual indent can underestimate the projected and contact area in case of "sink-ins" or in case when the height

of the piled-up material is less than 10% of the maximum height. The histogram analysis also works within similar margin of error.

The areas determined for the ROI by the above methods are: (i) surface area, (ii) projected area, (iii) contact area, where the contact area is expressed as a function of the projected area in case of microindentation and as a function of contact depth in case of nanoindentation. The hardnesses calculated from the three areas measured are: (i) Vickers Hardness (kg/mm^2) (ii) Contact Hardness (MPa) (iii) Indentation Hardness (MPa).

The indentation diagonal is calculated from the area of ROI measured by the above methods, while the residual indents depth and angles are determined from the AFM images of the microindentation case, and from the force-displacement graphs in case of nanoindentation.

An alternative method is to measure the diagonal directly off a line profile obtained by identifying corners of the residual indent from the 2-dimensional AFM image. This is a standard feature of commercial software for AFM image analysis. Fig. 1 (A) and (B) shows a typical AFM image of indentation in 3- and 2- dimensions respectively, while in Fig. 1(C) shows the region around one of the corner of the indent magnified. This method of measurement is subject to 3 possible sources of errors: (i) errors due to selection of the points defining the opposite corners of the indent, (ii) the line profile may not necessarily trace opposite edges of the indents, (as seen in Fig. 2) (iii) the length of the diagonal is also susceptible to error because of point (i).

To minimize these sources of errors a dedicated program was written in Matlab Ver 4.2.c1 and will be referred to as the AutoIndent program which provides a systematic method for the analysis of the indent [7]. By means of the AutoIndent program, the 'true' diagonal line profile can be traced along opposite edges of the residual indent passing through the apex (minimum height position point 'O') (see fig. 3). The AutoIndent program allows the analysis

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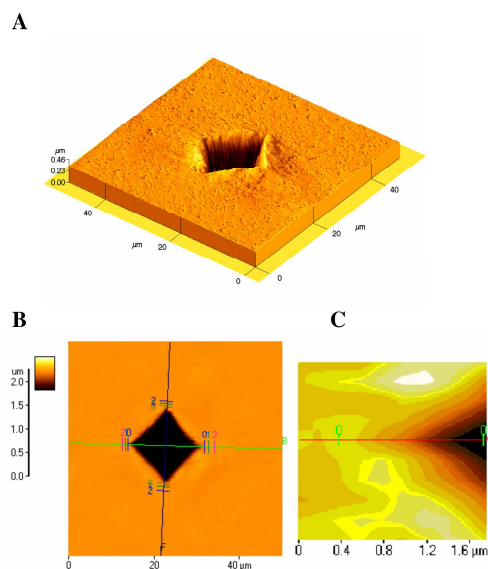


Figure 1. Shows a typical 3- and 2-dimensional AFM image of residual indent, (A) and (B) respectively. Also shown in (C) is the area around the corner of the indent magnified.

of the entire morphology of the residual indent and the piled-up material around the indent [3]. This paper presents a new method to determine the area of an indent by systematically and consistently identifying the edges of the residual indent and by obtaining a line profile along opposite edges. From this line profile one can measure parameters such as the diagonals, the depth of residual indent, the angles between opposite edges and the piled-up material. In addition this method allows complete characterisation of the elastic recovery of the indentation.

2 Method

The experimental work was carried out on WC 6 wt.% Co having a mean WC grain size of 1.1 μm. The Vickers microindentations were produced using a LECO M-400 testing machine, at indenting loads ranging from 50 gm to 1 kg. The residual indentations were measured using a PSI CP AutoProbe atomic force microscope in contact mode. The images were acquired in the 256 X 256 mode (i.e. data points). The images were preliminarily analyzed using the AFM image analysis

software ProScan. However, this program was found to be inadequate to give consistent answers, a problem experienced with most commercial software for AFM indentation image analysis.

2 AutoIndent

The AutoIndent program allows the automated analysis of an AFM image of the indent, characterizing the entire morphology of the residual indent and its surrounding piled-up material. The AutoIndent program consists of the following basic steps:

- (i) Determine the minimum point of the 3-D image of the AFM which is the apex of the indent or point 'O'.
- (ii) Define the indent by identifying the "square" boundaries [8] at regular height intervals of the residual indent (fig.2).
- (iii) Determine the corner points of each of the squares identified in (ii) (fig.2).
- (iv) A straight line is drawn from 'O' to corners points along each of the edges. (Fig 2)
- (v) The lines drawn in (iv) are combined generating the "true" line profile tracing the edges of the indent and are extended outside the indent on both sides of the AFM image.
- (vi) The line profile obtained is now used to measure parameters.

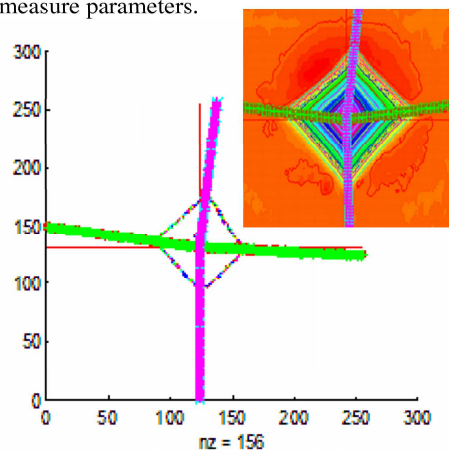


Figure 2. Corner points of the squares are used in defining the edge of the residual indent. The insert image shows a top view of the residual indent with the diagonal line profile (bold broad line) tracing the edges of the residual indent superimposed on it.

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A typical line profile obtained by AutoIndent is given in fig 3. The measurements from the AutoIndent can be divided into two categories: (a) Line profile measurements (e.g. diagonal, depth and angle). (b) Surface measurements (e.g. area, volume (obtained by defining ROI in a 2-D AFM image) and orientation). Steps (iii) – (vi) can be adapted to line profiles along the centre of opposite faces of the residual indent. A line profile along the centre of opposite faces of the indents is given in fig 4.

3 Line Profile measurements

3.1 Diagonal

The “diagonal line profile” obtained by the program AutoIndent is a trace along opposite edges of the residual indent (see Fig. 2 insert) passing through the apex (minimum position point) ‘O’ Fig. 3 shows a typical line profile obtained by AutoIndent.

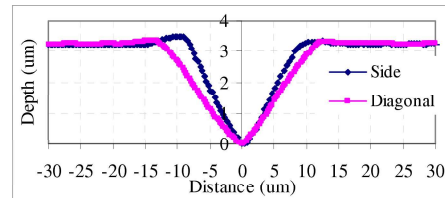


Figure 4. Line profile along the edges and centre of the faces of the indentation.

The line profile given in Fig. 3 shows that three pairs of distinct points (T and T', M and M' and I and I') are identifiable with a fourth point marked 'O' corresponding to the apex of the indent. The two pairs of points, T and T' and I and I', were determined by the gradient method [9], while the third pair of points, M and M', corresponds to the maximum vertical distance from point 'O'. Note should be made that:

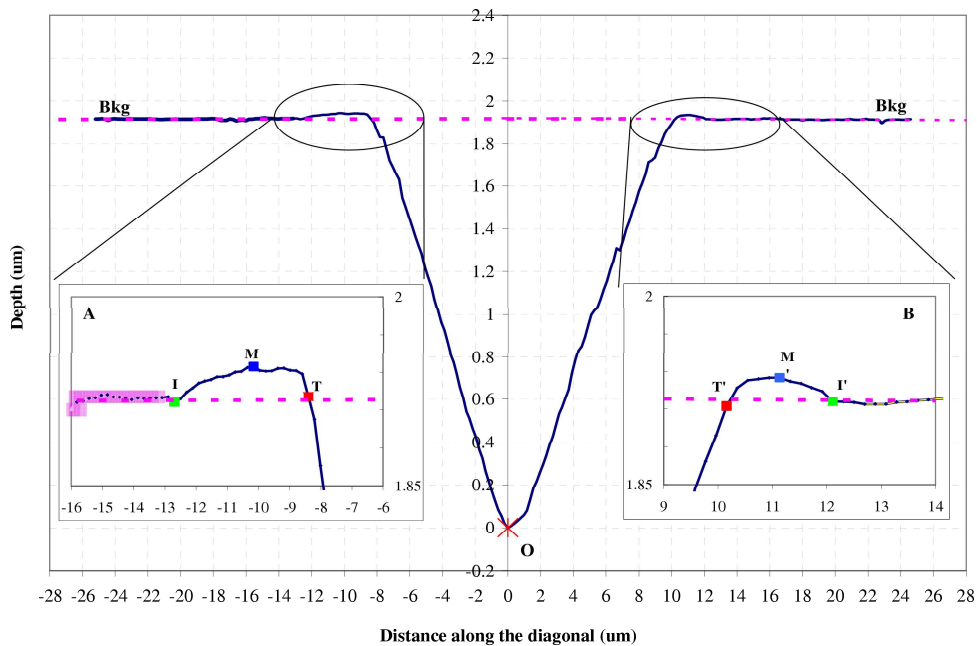


Figure 3. Line profile along opposite edges of a residual indent obtained by AutoIndent. The y-axis gives height with respect to the minimum point 'O'. The 'V'-part of the line profile is along the edges of the residual indent, while the horizontal dotted line represents the 'original' surface. Points T, T', M, M', I and I' are defined in text. The line profile shows piled-up material on both sides of the indent, which is magnified in the inserts A and B. (Note: the scale on the x- and y-axes is not the same).

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- (a) Although the points T and T' were obtained by the gradient method, in some cases they were re-evaluated by the median filtered differencing method [10]. When piled-up material is present, these points define the boundary between the residual indent and the level of the original surface.
- (b) The points I and I' were always determined by the median filtered differencing method [10]. These points were assumed to be the boundary between the piled-up material and the original surface level, since this boundary is not well defined

Fig. 5 gives the distances TT', MM' and II' for WC 6 wt.% Co sample. It has been shown by the authors that the length of the diagonal between II' gives hardness within 5% of hardness quoted by manufacturers of the carbide samples [7].

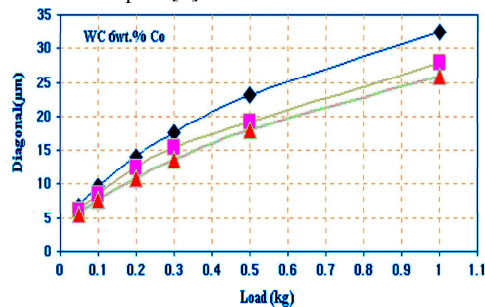


Figure 5. Distances between points (▲)TT', (○) MM' and (□) II' for WC 6wt.% Co samples at the loads tested.

3.2 Depth and Angle

Two additional parameters that can be determined by AutoIndent are depth and angle. The residual indentation depth, i.e. the vertical distance between the 'original' surface and point 'O' in fig. 3, is easily measured. The vertical height between M and I and between T and M allows the characterization of the piled-up material around the indent. From the three dimensional AFM image, the apex angle of the residual indent was determined as the angle TOT' between the straight lines joining 'O' to T and 'O' to T', although it was found by AFM that the inner surfaces and edges of the indentation are not plane but are convex. A significant difference of 5 – 8 % is observed, between these measured angles and the angle of

a Vickers indenter between opposite edges (148.8°) is observed, despite a large scatter.

4 Surface measurements

An AFM image provides 3-dimensional information about the surface topography. In the case of the AFM image of the residual indent the topographical information can be divided into three categories (i) the shape and geometry of the residual indent, (ii) size, shape and extent of the piled-up material adjacent to the indents and (iii) original surface region away from the indent and from the piled-up material. To measure any surface feature, the region of interest (ROI) is selected by demarcating the feature on the 2-dimensional image or/and by defining the height threshold. Within the selected region the parameters of interest that have been measured are (i) surface area (ii) projected area (iii) surface roughness (iv) volume.

4.1 Area and roughness

In most commercial AFM analysis software the surface area of the ROI is calculated by assuming the area between four neighbouring points to be twice the area of triangular planes formed by connecting a point with two of its nearest neighbours, taking into account the height difference between the three points. The total surface area is twice the sum of all the triangular areas for the data points within the ROI. On the other hand, the AutoIndent program sums all the triangular areas in the ROI, rather than simply doubling the area as done by commercial AFM analysis software. The difference in the surface area measured for the residual indents between the commercial AFM analysis software and AutoIndent was observed to be < 5%.

The most frequently used way to express roughness for ROI is as the root-mean square (RMS) roughness. In the analysis of the indentations this is calculated for the surface far away from the indent, and it is taken into account when calculating the volume of the piled-up material. The root-mean square roughness is a standard feature of most AFM software's. In the case of WC-Co the order of magnitude of the RMS roughness is 4nm for WC 6wt.%Co.

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4.2 Volume

The measurement of the volume of a ROI is included in the “surface measurements” because the volume is calculated by most AFM analysis software by multiplying the projected area of the selected ROI by the average pixel height within the ROI relative to the minimum height of the entire image except when measured by either the “water-filling” or the “valley filling” method.

The algorithms for the volume of the residual indent and the piled-up material are different in the AutoIndent program. The arbitrary selection of the ROI by threshold is overcome by introducing a systematic and consistent method of defining the boundaries of residual indent and the piled-up material around it. The upper boundary of the residual indent is determined by the best fitting plane defined by the four points T and T's. The reference height z_{RI} of this plane is assumed to be the average vertical distance of all the points T's from point 'O'. The volume of the residual indent V_{RI} is calculated as

$$V_{RI} = A_P \sum_{i=1}^{N_{RI}} (Z_{RI} - Z_i) \quad (1)$$

where N_{RI} is the number of pixel points within the residual indent, the area of each pixel A_P is $(S/256)^2$, where S is the scan size, and Z_i is the height of the i^{th} pixel point. The volume of the residual indent and the piled-up material around the indent is given in fig. 6. The volume of the piled-up material is corrected for by subtracting the volume contribution due to the surface roughness within the ROI (equation 2).

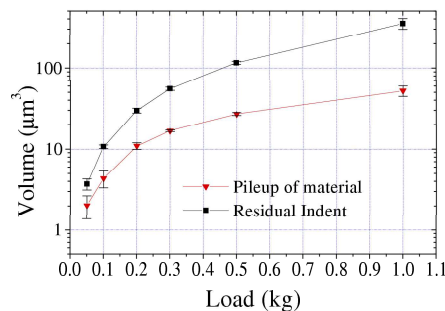


Figure 6 Volume of the residual indent and piled-up material for WC 6 wt.% Co.

4.3 Orientation

A novel way of observing the recovery of the material especially along the sides of the indent is by representing the region in terms of spherical coordinates. This method can be used to identify the edges of the indent and characterise the recovery and surface rotation (change of orientation) of the material.

In fig. 7 the angle between the plane parallel to the 'original' surface containing the apex point 'O' and the straight line joining point 'O' to each of the data points in the AFM image is shown. Fig. 7 was generated by the AutoIndent. For the Vickers indenter, there are two angles of interest: (i) 136° angle between opposite faces, (ii) 148.1° angle between opposite edges. The faces of the Vickers indenters are plane as observed by Hasche et al. [11]. Each of the data point of AFM images can be expressed in terms of spherical coordinates with point 'O' (minimum height point) of the residual indents' as its origin. Of relevance to this work is the azimuthal angle ϕ . In fig 7, (a) is the AFM image of the residual indent and (b) is the azimuthal angle image for the same indent. Comparing images (a) and (b): the AFM image shows the size of the residual indent and its surroundings without any detailed information about the shape of the indent faces and edges. While the azimuthal angle image has four distinct features: (i) the dark dot in the centre is the apex point of the residual indent's AFM image (ii) four straight lines from the centre correspond to the edges of the residual indent. (iii) between the edges is the region of varying contrast which represents the faces of the residual indent, (iv) the region of concentric rings corresponds to the region surrounding the residual indent.

In the 2-dimensional azimuthal angle image (Fig. 7 b), the edges are easily identified as are indicated by the solid arrows, the region between the edges for each of the quadrants marked A, B, C and D shows a non-uniform contrast, a feature not easily visible in the residual indent AFM image.

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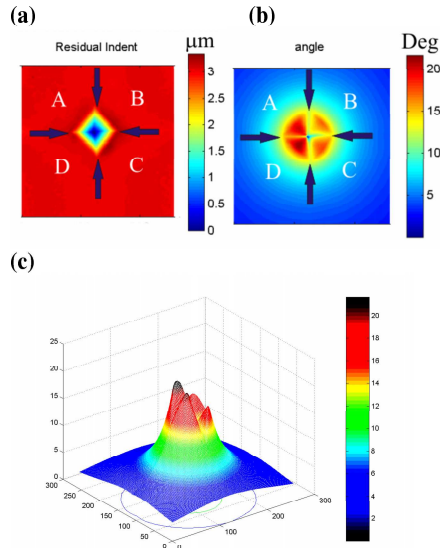


Figure 7: (a) is the AFM image of a residual indent, while (b) is the azimuthal angle image with respect to minimum height point 'O' of the residual indent. The solid arrows indicate the direction along the edges of the indent and letters A, B, C and D refer to the faces of the indent. (c) Azimuthal angle image - the points of maximum angle along each faces are the peak points or maximum angle in 3-D.

A three dimensional representation of the azimuthal angle (Fig. 7 (b)) is given in Fig. 7(c) shows four peaks for each of the faces of the residual indent. The peaks show that there is non-uniform recovery of the material and the point of maximum azimuthal angle i.e. is the highest point in each of the peaks. This point corresponds to the point of maximum curvature for each of the faces of the AFM residual indents' image. The valleys between the peaks are along the edges of the residual indent. The variation in the peaks height may be attributed to non-homogenous material.

4.4 Piled-up material measurements:

The pileups (or piled-up material) are defined as the material that rises above the original surface during the loading and unloading of the indenter. The ROI considered for this calculation is assumed to be annular with the outer radius being up to 20% longer than the

diagonal of the indentation. The volume of the piled-up material is corrected for the contribution of the surface roughness. The volume of the pileup of material V_{PU} is given by

$$V_{PU} = A_P \sum_{i=1}^{N_{PU}} (Z_i - Z_{bkg}) - N_{PU} A_P Z_{rms} \quad (2)$$

where N_{PU} is the total number of pixels within the ROI, Z_{bkg} is the average height of the original surface and Z_{rms} is the RMS roughness of the original surface.

The pileup can further be characterised in terms of the height and length. The points T, M and I identified on the line profile in fig.3 can be used to define the shape and size of the piled-up material. The parameters that can be used are: (a) The ratio between the distance II' and TT', ($L_{II'}/L_{TT'}$). (b) The total horizontal distance between points T-M and T'- M' (L_{TM}), and M-I and M'-I' (L_{MI}). The piled-up material can be analyzed in terms of its length L_{TI} (the total horizontal distance between points T-I, and T'-I'), which is equal to $L_{TM} + L_{MI}$. The shape can be analysed in terms of the ratios of these lengths L_{MI}/L_{TI} and $L_{MI}/L_{TT'}$, as shown, as an example, in Fig. 8 for a WC 6 wt% Co sample.

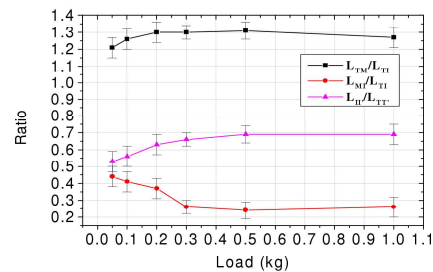


Figure 8. Shape and size of pileups for WC 6wt. % Co.

Conclusion

This paper has shown that a complete topographic description of a residual microindent and of the surface surrounding the indent can be given by means of the program AutoIndent. In addition, AutoIndent has the capability to observe changes in orientation. Results for WC 6wt. % Co. has shown the dependence of microindentation features on sample composition and indenting load.

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The size effect in microindentation

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The theory by Stelmashenko *et al.*, De Guzman *et al.* and Nix and Gao of the size effect in microindentation is outlined and some implicit assumptions are discussed. By an algebraic rearrangement, the slope of the straight-line plot of the square of hardness against the reciprocal of the diameter or depth of an indentation acquires a value which is predictable and in order of magnitude in agreement with experiment. The corresponding scale length, of order 1 micron in copper, is thus a real physical property of the material, although it is not visible in the undeformed material. In the recent experiments of Motz *et al.* it is observed directly. The two-phase 'hard metal' WC-Co has a microscopic intrinsic scale length, accordingly, the plot is no longer linear.

1. Introduction

We consider the indentation of a nominally homogeneous medium by a rigid indenter in the form of a cone of arbitrary cross-section. Since there is no scale length, the hardness, defined as the ratio of the load to the area of the indentation, must be independent of the size of the indentation. This is true whatever the elastic–plastic properties of the medium.

In fact, every medium has an intrinsic scale length, the interatomic spacing b , of order 3×10^{-10} m. Real materials contain dislocations with an areal density ρ , which implies a characteristic distance $\rho^{-1/2}$ between neighbouring dislocations. The dislocation density ρ usually lies between 10^8 and 10^{15} m⁻² with a corresponding separation between 10^{-4} and 3×10^{-8} m. Indentations with diameters between about 10^{-8} and 10^{-7} m are usually described as nanoindentations; those with diameters 10^{-6} m or somewhat larger are called microindentations. The indenters used in practice are cones of large angle and the depth h of the indentation is about a tenth of the diameter $d = 2a$.

On the scale of a nanoindentation there would normally be no dislocations in the region of concentrated stress within h of the tip of the cone, and the

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deformation is purely elastic and reversible. As the indentation proceeds, this region of concentrated stress expands until either it includes an existing dislocation or it is large enough for dislocations to nucleate in the homogeneous crystal. Dislocations then multiply. This may occur so rapidly that the load required to maintain the imposed rate of indentation falls despite the increase in the size of the indentation.

On the scale of a microindentation $d \geq 10^{-6}$ m, plastic deformation is spreading from a region which is heavily disturbed with a dislocation separation of the order 10^{-8} m. The medium is now an effectively homogeneously dislocated material. The dislocation separation is no longer the scale length, which is now b , the Burgers vector.

2. The current model

The current model was first analyzed by De Guzman *et al.* [1], and developed in detail by Nix and Gao [2]. Essentially the same results, with somewhat different numerical coefficients, had previously been published without derivation by Stelmashenko *et al.* [3]. We shall follow the details of Nix and Gao.

The indenter is assumed to be a rigid circular cone of half-angle θ (figure 1). The diameter of the indentation is $2a$, its depth h . Hence

$$h = a \tan \theta. \quad (1)$$

In the case of a homogeneous deformation, the critical resolved shear stress τ on a glide plane is given in terms of the dislocation density ρ_s by

$$\tau = \alpha \mu b \rho_s^{1/2}, \quad (2)$$

where α is taken to be $1/2$ and μ is the shear modulus.

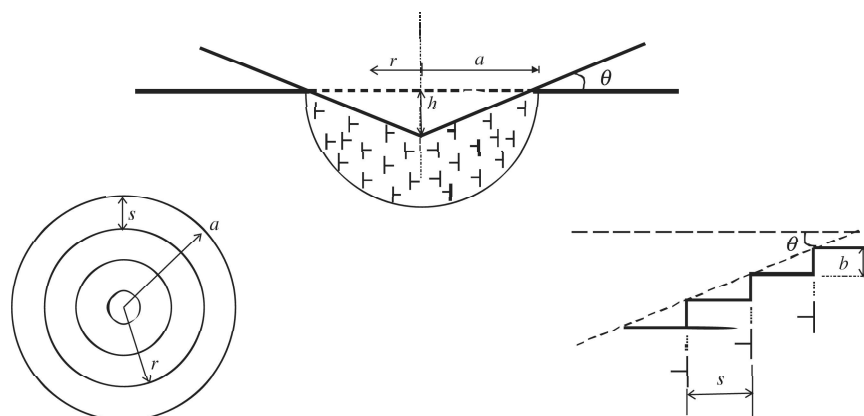


Figure 1. The indentation model of De Guzman *et al.* [1] and Nix and Gao [2]. Indentation by a rigid circular cone proceeds by the glide of circular edge dislocations normal to the surface.

Here, ρ_s is the density of ‘statistically stored’ dislocations. When the deformation is inhomogenous, involving either a strain gradient or a curvature k (of dimensions of L^{-1}), there is also a density ρ_g of geometrically necessary dislocations (GNDs) given in order of magnitude by

$$\rho_g = k/b. \quad (3)$$

The value of ρ_s at any point in a small indentation is taken to be the dislocation density at the geometrically corresponding point in a large indentation. It is assumed that the critical resolved shear stress is then given by

$$\tau = \alpha\mu b(\rho_s + \rho_g)^{1/2}. \quad (4)$$

For a polycrystal, the tensile flow stress σ is given by the von Mises relation to be

$$\sigma = \sqrt{3}\tau. \quad (5)$$

The hardness H is given by

$$H = 3\sigma = 3\sqrt{3}\tau = 3\sqrt{3}\alpha\mu b(\rho_s + \rho_g)^{1/2}. \quad (6)$$

The hardness of a large indentation is

$$H_0 = 3\sqrt{3}\alpha\mu b(\rho_s)^{1/2}. \quad (7)$$

We now estimate ρ_g . It is assumed as in figure 1 that the plastic deformation during indentation proceeds by simple prismatic punching, loops of dislocations being equally spaced along the surface of contact. Then the spacing s is given by

$$s = b \cot \theta. \quad (8)$$

The length of a dislocation loop of a radius r is $2\pi r$, and the number of loops between r and $r + dr$ is dr/s . The total length λ of dislocation line is thus

$$\lambda = \int_0^a \frac{2\pi r dr}{b \cot \theta} = \frac{\pi a^2 \tan \theta}{b}. \quad (9)$$

The plastically deformed region is assumed to be a hemisphere of radius a and volume

$$V = \frac{2}{3}\pi a^3. \quad (10)$$

The density of geometrically necessary dislocations is thus

$$\rho_g = \frac{\lambda}{V} = \frac{3 \tan^2 \theta}{2bh}. \quad (11)$$

It follows from (6), (7) and (11) that

$$\left(\frac{H}{H_0}\right)^2 = 1 + \frac{h^*}{h}, \quad (12)$$

where

$$h^* = \frac{81}{2} b \alpha^2 \tan^2 \theta \left(\frac{\mu}{H_0}\right)^2. \quad (13)$$

Nix and Gao showed that the experimental observations both on the (111) face of a single crystal of copper and on cold-worked polycrystalline copper gave remarkably accurate linear relations between $(H/H_0)^2$ and $1/h$. They noted that h^* is not a constant for a given material and indenter geometry, rather it depends on the statistically stored dislocation density through H_0 .

The argument is geometrically robust. If figure 1 is taken to represent indentation by a wedge rather than by a circular cone, the length of geometrically necessary dislocation line per unit length of indentation is

$$L = \int_0^a \frac{2dr}{b \cot \theta} = \frac{2a \tan \theta}{b}, \quad (14)$$

while the plastically disturbed area is

$$A = \frac{1}{2} \pi a^2, \quad (15)$$

leading to a geometrically necessary dislocation density of

$$\rho_g = \frac{L}{A} = \frac{4 \tan^2 \theta}{\pi b h}, \quad (16)$$

differing little from (11).

3. Some assumptions of the model

Equation (4) is clearly a crude approximation. The dislocation density tensors for statistical and for geometrically necessary dislocations are not isotropic, but display the geometry of the strain and the strain gradient respectively. Their interaction depends on the slip systems actually available (e.g. in fcc or bcc structures).

The prismatic dislocation loops of figure 1 represent the most efficient way of meeting the geometrically necessary dislocation requirements. Dislocations of the necessary glide system will generally not be available, and the actual density of geometrically necessary dislocations will exceed that given by (9) by a factor of order $\sqrt{3}$.

It is assumed in figure 1 that the plastically deformed region is a hemisphere of radius a . The observations of Dyer [4] for the indentation of the (100) face of a single crystal of copper by a sphere show heavy concentration of dislocations in a region which is roughly a sphere of radius $3a$, so that V would exceed that given by (10) by the large factor of 54. The survey by Samuels [5] shows that

the geometry of the heavily deformed region depends on the form of the stress–strain curve, specifically for an ideal elastic–plastic model on the value of $(E/Y)\cot\theta$, where E is Young’s modulus and Y the yield stress.

Motz *et al.* [6] have measured the local crystallographic orientation change due to the indentation under the indenter flank in copper grains, showing the misorientation distribution to be extended up to $2a$. The orientation gradient associated with the strain gradient caused by GNDs is $1.8^\circ/\mu\text{m}$ or $31.4\ \mu\text{m}$ per radian. Durst *et al.* [7] have considered the plastically deformed volume underneath the indenter by approximating the size of the plastic zone at an equivalent plastic strain of $\approx 1.5\%$, using the contact radius to be $fa = 1.9a$; the density of GNDs is then $1/f^3$ of the value (10).

These two considerations suggest that ρ_g is usually about $\sqrt{3}/10$ times the value given by (11), and h^* correspondingly smaller by a factor of about 0.67.

The model implicitly assumes that the statistically stored and geometrically necessary dislocations are distributed similarly in space. This may well not be true. However, the consideration is not relevant in the present approximation which considers only mean dislocation densities and adds them linearly.

Stilwell and Tabor [8] showed that, when a conical indenter is removed from an indentation in steel or a commercial alloy, the recovery in depth is almost purely elastic. Nevertheless, the loops of dislocation in figure 1 represent a dislocation pile-up which has resisted the initial plastic indentation. This kinematic hardening implies that the increase in hardness produced in a small indentation by the geometrically necessary dislocations is greater than that implied by (4), and the value of h^* correspondingly larger. This partially compensates the reduction in h^* estimated from geometrical considerations.

4. Extension of the model

Equation (12) predicts that the plot of $(H/H_0)^2$ against $1/h$ will be a straight line, but does not predict the slope of the line. If (12) is rewritten as

$$H^2 = H_0^2 + \frac{81}{2}\alpha^2 b\mu^2 \tan^2\theta \frac{1}{h}, \quad (17)$$

the slope of the plot of H^2 against $1/h$ is determined by the numerical factor $81\alpha^2/3$, the material constant $b\mu^2$ and the geometrical factor $\tan^2\theta$ of the indenter. This provides a quantitative test of the model. Nix and Gao assumed $\alpha = \frac{1}{2}$; the usual value is $\alpha = \frac{1}{3}$. The experimental slopes shown for copper in figures 3 and 4 of Nix and Gao are compared in table 1 with those predicted by (14) with the values $b = 2.56 \times 10^{-10}\text{ m}$, $\mu = 42\text{ GPa}$, $\tan\theta = 0.358$ and $\alpha = \frac{1}{2}$ or $\alpha = \frac{1}{3}$. The agreement is satisfactory.

The analysis by Fleck *et al.* [9] of torsion experiments on cold-drawn copper wires led to values of h^* between 2.6 and $5.1 \times 10^{-6}\text{ m}$, with a mean of $3.7 \times 10^{-6}\text{ m}$. The analysis by Stölken and Evans [10] of bending tests on thin foils of polycrystalline copper led to values in the range $3\text{--}5 \times 10^{-6}\text{ m}$.

A further check is possible. The experiments analyzed by Nix and Gao were performed in a range of hardness centred around 1.3 GPa , corresponding to a resolved shear stress of $1.3/3\sqrt{3} = 0.25\text{ GPa}$. The survey by Basinski and Basinski [11]

Table 1. Comparison of predicted and measured values of the characteristic length h^* for values of $\alpha = \frac{1}{2}$ and $\frac{1}{3}$.

Material	H_0 (GPa)	h^* (μm)	μ (GPa)	b (nm)	α	h^* (predicted) (μm)
(111) Single crystal Cu (annealed)	0.581	1.60	42	0.256	1/2	1.73
	0.581	1.60	42	0.256	1/3	0.77
Polycrystalline Cu (cold worked)	0.834	0.464	42	0.256	1/2	0.840
	0.834	0.464	42	0.256	1/3	0.373

of dislocation densities in single crystals of copper deformed in tension shows a corresponding dislocation density of about 10^{-12} m^{-2} , with a separation between statistically stored dislocations of $1 \times 10^{-6} \text{ m}$. In the present theory, h^* is the reciprocal of the strain gradient at which the separations of neighbouring geometrically necessary dislocations and of neighbouring statistically stored dislocations are equal. The agreement is again satisfactory.

The existence of a characteristic length of the order of a micron in copper strained by about 10% seems well established. The length has no significance in the perfect copper crystal; it appears only on plastic deformation. It arises from a balance between the dislocation densities associated with homogenous plastic strain and with plastic strain gradient. Curiously, the theory of the latter, treated as a perturbation, is simpler than the theory of the former.

5. Direct observation of the characteristic length

Although the existence of a characteristic length in the plastic deformation of copper has been demonstrated, its value has been derived from an analysis of mechanical measurements. A direct observation has recently been published by Motz *et al.* [6]. It is first necessary to recall that a component of strain such as $e_{ij} = (\partial u_j / \partial x_i + \partial u_i / \partial x_j)$ and a component of rotation such as $\omega_{ij} = \frac{1}{2}(\partial u_j / \partial x_i - \partial u_i / \partial x_j)$ are both linear combinations of components of the same displacement gradient tensor, and that their gradients such as $\partial e_{ij} / \partial x_k$ and $\partial \omega_{ij} / \partial x_k$ are quantities of the same kind. Motz *et al.* made Vickers indentations of diameters 3.3–16.3 microns in grains of polycrystalline copper of mean grain size 150 microns, thus effectively single crystals of random orientation. The indentations were sectioned normal to the surface, and the lattice rotations were measured by electron-backscattering diffraction. They found that the regions of highest orientation changes are concentrated beneath the indent flanks and below the indent tip. The rotation patterns did not scale with the size of the indentation. Instead, the width of the regions of maximum misorientation were constant at about 1 micron, whilst the ratio of the maximum misorientation to the width of the Vickers imprint was constant at about 1.8 degrees per micron. This corresponds to a radian in 31.8 microns of indentation diameters. The principal rotation gradient is in depth, and the gradient in depth is a radian in 4.5 microns, larger than but of the same order as other estimates of h^* .

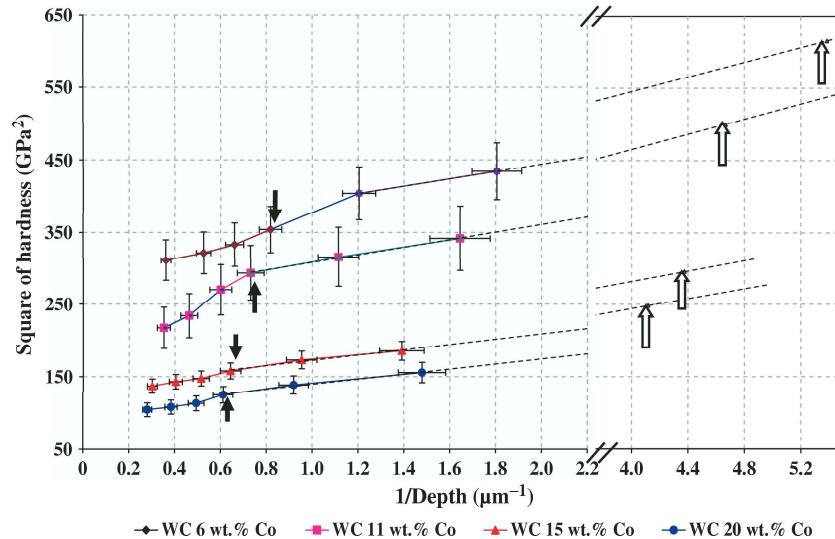


Figure 2. The square of the hardness as a function of the reciprocal of the depth of the Vickers indentation for samples of WC-Co hard metal with four different weight percentages of cobalt binder. The filled vertical arrows indicate the stages of indentation at which the depth h of the indentation is equal to the intrinsic characteristic length of the sample; the open arrows indicate the stages at which the width of the indentation is equal to the characteristic length.

6. A medium with an intrinsic length scale

If a medium has an intrinsic length scale, it is to be expected that the plot of H^2 against $1/h$ will no longer be a straight line but will show an anomaly when h is of the order of this intrinsic length. 'Hard metal', consisting of grains of hard tungsten carbide WC cemented together with relatively soft cobalt, is such a medium. Four grades of hard metal were studied, each with a carbide grain size of about a micron, but with 6, 11, 15 and 20 weight percent of cobalt, and correspondingly large volume percentages. The scale length was taken to be the sum of the carbide grain size and the mean free path in the cobalt. The results are shown in figure 2, where the vertical arrows mark indentations of a depth equal to the characteristic length, 1.30, 1.41, 1.52 and 1.69 microns for the four materials.

The physical interpretation of these observations is still to be determined. A tentative model is that when h is greater than the critical length, the medium is effectively a homogenous elastic-plastic material. When h is less than the critical length the deformed layer is resting on an elastic substrate. Even for indentations deeper than the critical length, it is unlikely that the present theory will apply. The very rapid work hardening of hard metals seems [12] to depend on a mechanism different from the dislocation forest-cutting mechanism on which (2) is based. There is no reason to believe that the contributions to the flow stress from

macroscopic hardening and from the geometrically necessary dislocations will add quadratically as is assumed in (6) and (12).

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The effect of elastic relaxation on the indentation size effect in tungsten carbide

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The Vickers hardness (HV) of a material is measured in terms of the length of the diagonals of the indentations produced when a square pyramidal diamond indenter is loaded into the material. HV is defined as the ratio between the indenting load and the area of the indentation visible after unloading the indenter:

$$HV = 2 \sin \alpha \frac{P}{d^2} \quad (1)$$

where α is the apex half-angle of the pyramidal indenter, P is the indenting load in kgf and d is the indentation diagonal in mm. The diagonal, d , is measured by means of an optical microscope after unloading the indenter, while the apex half-angle of the indentation, α , is assumed to be constant at all applied loads and equal to the apex half-angle of the indenter, i.e., half of the angle between opposite triangular faces of the indenter (standard $\alpha = 68^\circ$).

It is well established that in most materials the microhardness measured by means of indentations decreases with increasing indenting load down to a constant value, which coincides with the macrohardness of the material [1]. This phenomenon is called indentation size effect (ISE) and has been observed in many materials, including tungsten carbide (WC) single crystals [2].

This work aimed at assessing the role of elastic relaxation in the observed ISE. For this purpose, a (0001) face of a WC crystal was indented at 25, 50, 100, 200 and 500×10^{-3} kg and, after unloading the indenter, the depth and diagonal lengths of the indentations were measured by atomic force microscopy (AFM). The results were compared with the depth and diagonal of the indentations before unloading, which were calculated from the geometry of the indenter.

The indenter was a standard Vickers indenter, i.e., a square pyramid of apex half-angle equal to 68° . For the calculation of the size of the indentation before unloading, it was assumed that the intrinsic hardness of the WC (0001) surfaces was 2000 kgfmm^{-2} , i.e., the macrohardness measured at loads of 1 kg or higher [3].

Fig. 1 shows the percentage change in the depth and the diagonal length of the indentations before

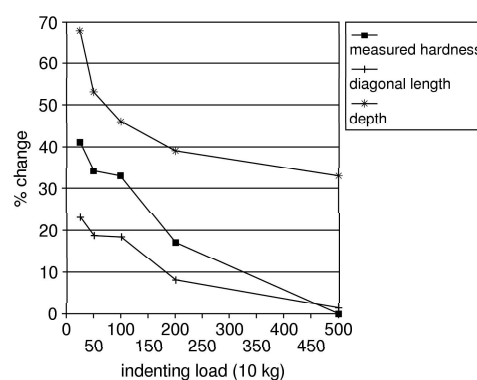


Figure 1 Plot of the percentage change in the measured hardness with changing indenting load and of the percentage change in the diagonal length and depth of indentations at various indenting loads in a (0001) face of a WC crystal, before and after unloading the indenter.

and after unloading each of the five indenting loads, as well as the percentage change in Vickers microhardness, relative to the known macrohardness. The percentage change decreases in all cases with increasing indenting load. The microhardness tends to the macrohardness value, and the percentage change in diagonal length tends to zero, but the change in depth tends to approximately 30%. The half-angle β between two opposite edges of the indentation, which, before unloading, is related to the apex angle α by the equation

$$\tan \beta = \sqrt{2} \tan \alpha \quad (2)$$

and measures 74° before unloading but is larger than 74° after unloading. Table I lists the values of both α and β at each indenting load, as measured by AFM after unloading, and the percentage changes in the angles, relative to their values before unloading. After unloading, α and β are no longer related by Equation 2 because the relaxation of the faces and of the edges of the indentations is not a simple translation but involves bowing by different amounts.

Therefore, when measuring the Vickers hardness

TABLE I Values of the α and β half-angles as measured by AFM after unloading and percentage difference between these and the values before unloading

Indenting load (10^{-3} kg)	Measured β (after unloading)	Percentage change in β after unloading	Measured α (after unloading)	Percentage change in α after unloading
25	81.0°	9.5	76.0°	11.8
50	78.2°	5.7	74.8°	10.0
100	76.3°	3.1	71.7°	5.4
200	76.6°	3.5	70.6°	4.6
500	76.4°	3.2	71.1°	4.5

of a material, the angle α in Equation 1 should be measured and not assumed to be constant. Table II lists the H68 hardness values of the WC (0001) faces, i.e., the values obtained from the measured diagonals (measured by AFM after removing the indenter) and considering α constant and equal to 68° (as is done in practice), as well as the $H\alpha$ hardness values, i.e., the values obtained from the same diagonals but using the angles measured after unloading. Table II shows that the microhardness of the crystal, as defined by Equation 1, would be even higher than it was believed until now if the correct angle α were used.

The results given above show that indentations produced on (0001) surfaces of WC crystals by Vickers indenters relax partially when the indenter is unloaded, so that the depth of the indentations is at least 30% lower than the depth before unloading the indenter. While the change in length of the diagonals due to elastic relaxation is much smaller than the

change in depth, it results in measured hardness values that are substantially higher than the values one would obtain if no relaxation occurred. The change in the diagonal length due to elastic relaxation tends to zero (and thus the microhardness value tends to the macrohardness value) as the applied indenting load tends to 500×10^{-3} kg.

The elastic relaxation as measured by the change in the depth of the indentation does not tend to zero as the load tends to 500×10^{-3} kg, but it tends to a constant value, as the hardness does. This indicates that the amount of elastic relaxation allowed by the plastic deformation induced by the indentations plays a major role in the observed indentation size effect.

Acknowledgments

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TABLE II Microhardness values of the (0001) surface of a WC single crystal, calculated using $\alpha = 68^\circ$ (H68) and using the values of α derived from Table I ($H\alpha$)

Indenting load (10^{-3} kg)	H68 (kgfmm^{-2})	$H\alpha$ (kgfmm^{-2})
25	3408	3649
50	3029	3224
100	3020	3197
200	2372	2514
500	2043	2164

Chapter 9

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