

Sintering Behaviour of High-Energy Ball-Milled Ti-Mg and Ti-Al-Mg Alloy Powders

Davison Nyabadza

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requirements for the degree of Master of Science in Engineering.

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DECLARATION

I declare that this research project is my own, unaided work. It is being submitted for the Degree of Master of Science in Engineering at the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree at any other university.

(Signature of Candidate)

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ABSTRACT

Low-density Ti-Mg and Ti-Al-Mg alloy specimens have been produced by sintering mechanically alloyed powders. Alloying titanium with low-density elements has been found to reduce costs while improving on properties such as strength. Very few researchers have sintered Ti-Mg based alloys, thus literature on these alloys is very limited. The powders used in this work had been milled for different times. Consolidation was carried out at 27 and 33MPa. The specimens were sintered in a tube furnace under argon, at temperatures ranging from 630 to 900°C for up to 10 hours. Higher green densities were achieved at 33MPa. A decrease in green density with increasing milling time was observed. No sintering occurred at 630°C. Most of the samples were soft which made surface preparation difficult. Sintering produced porous samples with some showing extensive and interconnected porosity. Reduced porosities were observed on samples consolidated at 33MPa and sintered at higher temperature (900°C). Ti-6Al-4Mg specimens recorded hardness values of up to 914HV. XRD analysis revealed dissociation of the solid solutions formed during milling and oxidation in other samples. The Mg content retained in the sintered samples ranged between 0.5 and 4wt%. Densities as low as 4.23 and 4.21g/cm³ were achieved in Ti-10Mg and Ti-20Mg samples respectively. These density results are promising since they are comparatively lower than those of most Ti-based alloys. Further work will aim to reduce porosity, oxidation and magnesium losses.

DEDICATION

To my sweetheart Eldah and my family

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CHAPTER ONE

INTRODUCTION

1.1 Background

Titanium (Ti) and its alloys are among important advanced materials because of their low density, attractive combination of high strength and toughness, corrosion resistance and high temperature properties [1-6]. They are finding increasing use in the aerospace and automotive industries where there is an increased need for stronger and lighter materials due to the need for improved performance (i.e. fuel efficient engines) and safety. Titanium alloys also find use in applications such as architecture, biomedical replacements (implants), marine and off-shore, desalination plants, vessels, sporting equipment and computer components [7].

There is a need to reduce both the cost and density of Ti (4.51g/cm^3) to help expand uses and find new applications. Alloying titanium with elements such as aluminium (Al), vanadium (V), and silicon (Si), has been found to reduce costs while improving on properties such as strength [8]. Currently, the most commonly used titanium alloy in engineering is Ti-6Al-4V [8].

The need to reduce density has arisen from the recognition that a *density reduction is 3-5 times more effective* in reducing the structural weight of aerospace vehicles than increases in strength and modulus [2]. The density can be reduced by alloying titanium with low-density elements such as aluminium, magnesium (Mg) and silicon [2, 4-5]. Magnesium has a low solubility in Ti and boils at a temperature much lower than the melting point of Ti [1]. This makes it impossible to alloy Ti with Mg using conventional methods such as melting and casting. Thus, non-equilibrium processing techniques namely, mechanical alloying (MA) and physical vapor deposition (PVD) have been tried [2].

In this work, MA was chosen because it is relatively cheaper and can be used for mass production of powders. An inert environment (vacuum or argon) is required for both MA and sintering since Ti is very reactive. Thus, test specimens for sintering will be produced through consolidation of these powders at two different pressures.

1.2 Objectives

The major objective of this work is to explore the possibility of sintering low-density or light-weight Ti-Mg based alloys that maintain a high Mg content after sintering. The study will investigate the effects of particle size distributions and compaction pressure on sinterability of these mechanically

alloyed powders. A lot of work has been done on the production of Ti-based alloys, but most of the work did not involve alloying Ti with Mg because Mg has a very low solubility in Ti under equilibrium conditions. Only a few researchers have produced Ti-Mg based alloys through both mechanical alloying and sintering. Thus literature on the sintering of Ti-Mg based alloys is very limited. Effects of parameters such as powder particle size and compaction pressure have not been investigated. Thus, effects of these parameters on green density will be examined. Sintering has been carried out at higher temperatures (up to 1100°C) but this resulted in high Mg losses through evaporation. Therefore, relatively lower temperatures will be used (between 630 and 900°C). This project will also report on sintered properties such as sintered density, hardness and phase constituency. Since Mg is more abundant than vanadium, its use as a replacement for vanadium is expected to lead to the reduction of the overall costs of Ti alloys.

CHAPTER TWO

LITERATURE REVIEW

2.1 Titanium and Its Alloys

Titanium (Ti) is a light metal with a density of 4.51g/cm^3 [2] and undergoes a phase transformation from a hexagonal close packed (h.c.p) to body centered cubic (b.c.c) structure at 882°C [9]. It is the ninth-most abundant element in the earth's crust (0.63% by mass) and occurs as rutile and ilmenite ores [9]. Titanium is a highly reactive metal with respect to normal atmospheric gases such as oxygen, nitrogen and hydrogen; thus processing of this metal is carried out under a controlled atmosphere such as argon or vacuum.

Titanium alloys have found wide applications, but the most common uses are: aerospace [10-12], automotive [13], medical [3, 13], architecture [13], and marine [10, 13]. A major factor limiting its applications is its high cost [5]. While titanium is relatively abundant, its components are relatively expensive because it is difficult to isolate: the commercial isolation processes, the Kroll and Hunter processes, are time consuming and expensive [9].

Although titanium can be used in the pure form, attractive properties have been obtained by alloying it with other elements. Alloying is one way of

reducing cost. Titanium and titanium based alloys are characterised by: good fatigue strength; corrosion resistance and biocompatibility; relative low modulus; low densities, which give high specific strength-to-weight ratios allowing for lighter and stronger structures [3]. The densities of titanium-based alloys range between 4.43 g/cm³ and 4.85 g/m³, depending on the alloying elements [10].

2.2 Classification of Titanium alloys

Titanium alloys are classified as alpha, beta or alpha + beta, where the alpha and beta are the possible phases in the alloys. These are discussed briefly in the following Sections. The phase compositions affect their areas of application. According to Froes [6], applications of titanium alloys can be separated into two categories: high strength, good fracture toughness and fatigue behaviour; and lower strength and corrosion resistant. Generally, the alpha-beta and beta alloys are used for the former application and the commercially pure grades for the latter use.

2.2.1 Alpha (α) Alloys

These are single phase h.c.p alloys formed by alloying Ti with elements (such as aluminium) which stabilize the alpha phase [14]. Alloys in this group are characterized by relatively low strength, but superior creep resistance and

thus are preferred for higher temperature applications of up to 600⁰C [6, 14]. Examples include Ti-5Al-2.5Sn (where Sn is tin) (compositions in wt. %), Ti-6Al-2Nb-1Ta-0.8Mo (where Nb and Ta are niobium and tantalum respectively) and Ti-8Al-1Mo-1V [14].

2.2.2 Beta (β) Alloys

Beta alloys have a bcc structure at room temperature. They contain transition elements such as vanadium, niobium, and molybdenum which reduce the temperature of the alpha to beta phase transition. Occasionally, they contain small amounts of alpha stabilizers which precipitate resulting in the strengthening of the alloy. Beta alloys have excellent forgeability, hardenability and respond readily to heat treatment. Alloys in this group include Ti-10V-2Fe-3Al and Ti-15V-3Cr-3Al-3Sn (where Cr is chromium) [14].

2.2.3 Alpha+Beta ($\alpha+\beta$) Alloys

Alpha+Beta alloys have compositions that support a mixture of α and β phases and may contain between 10 and 50 wt% beta phase at room temperature [14]. Ti-6Al-4V is the most common alpha+beta alloy: it is the most extensively used Ti-based alloy because it offers performance which far outweighs the cost of the alloy [9]. Alpha + beta alloys have good forgeability and their properties are controlled through heat treatment.

2.3 Conventional Production Methods for Ti-Based Components

Most commercial titanium alloys are produced by casting and ingot metallurgy (IM) [14-16]. Comparisons between casting and IM components can be found in **Reference 15**. In most cases, cast parts exhibit inferior mechanical properties to IM parts because of the inability to control the microstructures and porosity inherent to the casting process [12]. On the other hand, IM require conversion to the required geometry (i.e. plate, sheet, wire, etc.) [15].

In both instances, the reactivity of Ti is considered so as to ensure defect free components. The most important thing to note is that these two methods are applicable to alloys that can be produced under equilibrium conditions. In these methods, processing is carried out under a vacuum or argon atmosphere since titanium is very reactive.

Ti alloys can also be manufactured by powder metallurgy (PM) techniques. In these techniques, solid components are made from powders by compaction followed by sintering at an appropriate temperature [14, 17]. Sintered Ti alloy components have been found to possess a combination of a good microstructure (homogenous microstructure) and mechanical properties [14]. The most important advantage for PM is that it offers a huge advantage for alloy development, which is one of the objectives of this project. The processing of metal powders in the solid state can eliminate alloy chemistry

limitations, thus allowing for a wide variety of alloy systems to be synthesized. Other benefits of PM can be found in, among others, **References 12, 14, 17 and 18**.

2.4 Ti-Mg and Ti-Al-Mg Alloys

Ti-Mg based alloys are attracting increasing research attention because of the potential to further reduce alloy density. For example, a major goal in the development of improved aerospace materials is to reduce density while maintaining adequate mechanical property levels [19]. Mg is particularly attractive as an alloying element for this purpose because it has a low density (1.74 g/cm^3) [1].

The Ti-Al-Mg system is aimed at replacing the conventional Ti-Al-V in some of its applications, particularly medical implants. Use of Ti-6Al-4V components as medical implants is increasingly becoming contentious because V is toxic [3]. There is also the added advantage that replacing vanadium ($\rho = 6.0 \text{ g/cm}^3$) with Mg in Ti-Al-V alloys will reduce the density of the alloy and result in lighter components. Mg is 3.5 times lighter than V and it has been estimated that each 1 at% of Mg in solid solution has the potential to reduce density by 1% [2].

There are two main obstacles to the manufacture of Ti-Mg based alloys. The first obstacle is the low solubility of Mg in Ti, of about 0.7 at% Mg at 890°C

under equilibrium conditions [20] (**Figure 2.1**). The other obstacle is the vast difference in the melting points of Mg and Ti. Mg melts at 651°C and boils at 1091°C while pure titanium melts at 1668°C [1, 21-23]. These differences make it impossible to produce Ti-Mg based alloys by casting and IM. To overcome these two obstacles, a new economically feasible method that can allow the extension of alloy content levels is required. A method that has been attracting a lot of attention is mechanical alloying (MA).

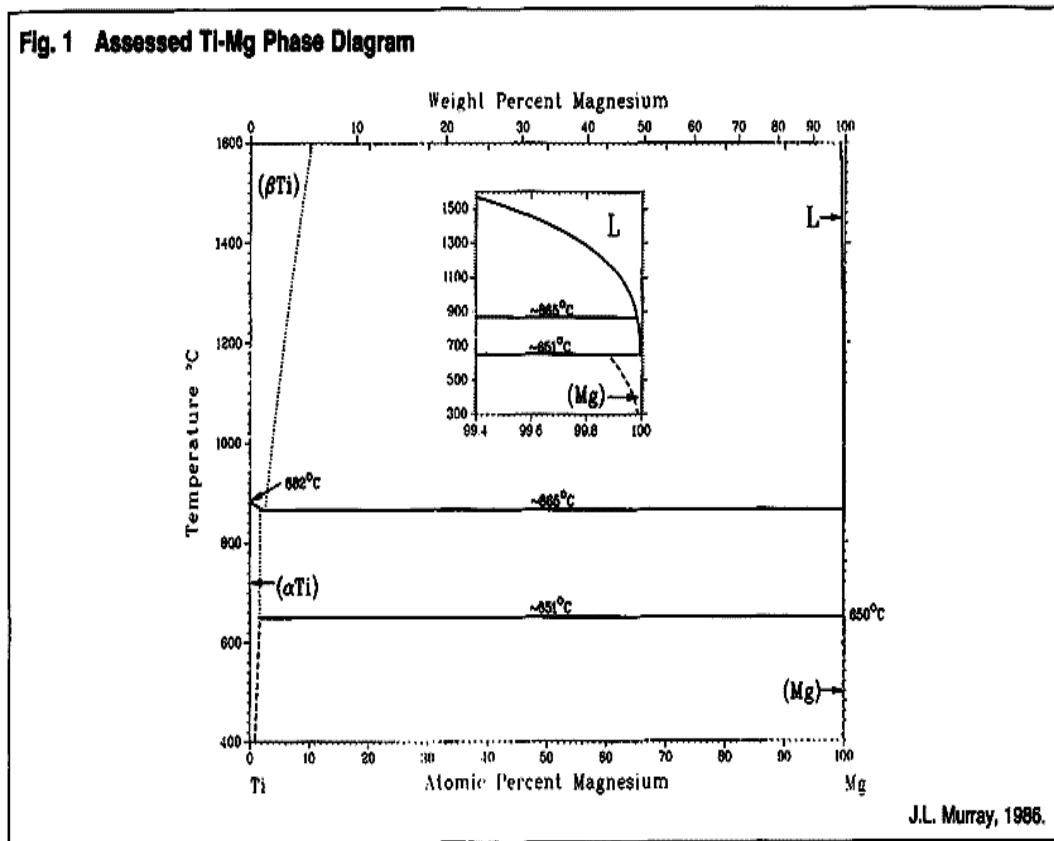


Figure 2.1: Ti-Mg phase diagram [21]

2.5 Mechanical Alloying

Mechanical alloying is a high energy ball milling technique which involves fracturing, welding and re-welding of powder particles resulting in the extension of solid solubilities and novel crystal structures. This method has been employed since the 1960s in the production of oxide-dispersion-strengthened nickel and iron-base super alloys [22]. It is an inexpensive method that can yield relatively large quantities of powders (up to 600 kg per day) that consists of nanometer-sized particles (usually less than 100 nm) [24-25]. The nanostructures formed during milling help extend solid solubilities [26-27].

Stages of powder evolution during mechanical alloying are shown in **Figure 2.2** [28]. New surfaces created in the early stages of milling enable the particles to weld together leading to an increase in particle size with some particles as large as three times more than the starting particles. The composite particles at this stage have a layered structure. With continued deformation, the particles become work hardened and eventually fracture by fatigue resulting in size reduction. At the same time, there is a rapid diffusion of atoms from one grain into the other, leading to quick homogenization and the formation of solid solutions [27].

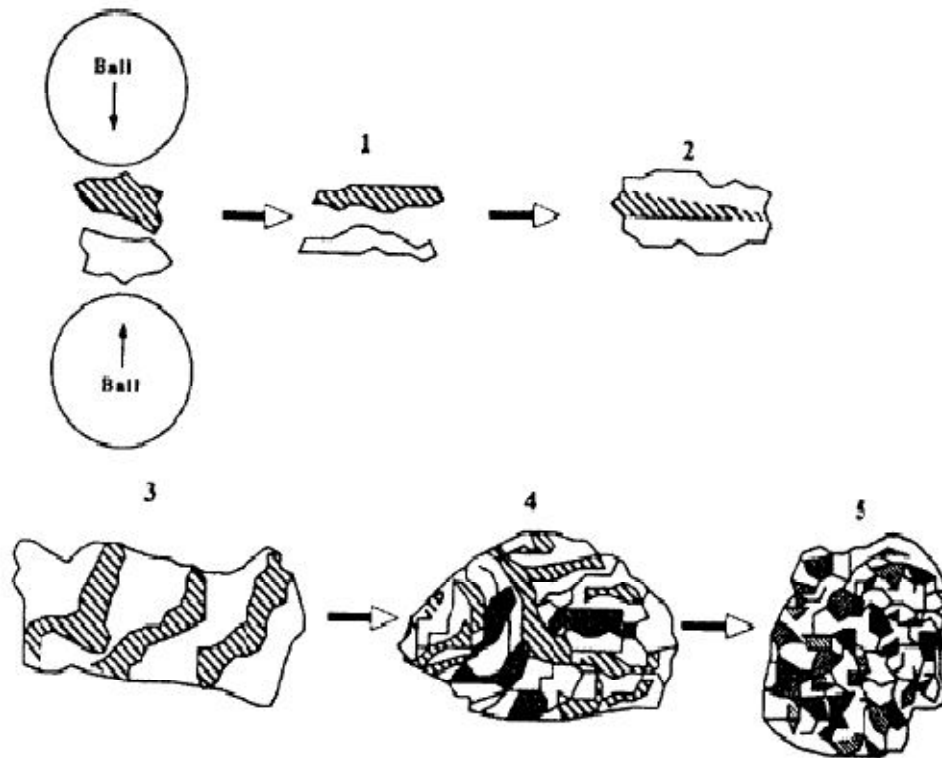


Fig 2.2: Stages of powder evolution during mechanical alloying of a mixture of ductile particles trapped repeatedly between two colliding balls [28].

Mechanical alloying has been successfully used for the synthesis of both Ti-Mg [1, 21, 23, 27] and Ti-Al-Mg [4-5] alloy powders. Sun and Froes [20] achieved a solid solubility of Mg in Ti of 24 at% (16 wt %) after 48 hours of milling in a high energy ball mill under an inert atmosphere (to eliminate contamination). However there is no information on the amount of powder yield that was obtained.

Small amounts of process control agents (PCAs), about 1 to 5 wt%, are added to minimize effects of cold welding on the particle during milling [27]. It is possible that the amount of PCA used by different researchers is influenced by the characteristics of the powders being milled, especially the particle sizes and particle size distributions. The PCA used should ideally be optimized on a case by case situation and the least amount possible used. This is because there is always an inherent chance of powder contamination by the PCA used [23].

MA is a complex process and hence involves optimization of a number of parameters to achieve the desired product phase and microstructure. The milling parameters include milling speed, milling time, ball-to-powder weight ratio (BPR), type and size of the grinding media, volume of load, milling atmosphere, process control agent (PCA) and type of mill .

As noted earlier, it is desirable to have a fine particle size, preferably nanosized, in order to have a high solid solubility of Mg in Ti.

2.6 Milling Parameters

2.6.1 Type of Mill

The mill determines the ease with which the required final powder constitution can be attained [22, 27, 29]. Mills that allow the temperature of the powder being milled to increase uncontrollably produce powders with a high degree of amorphotisation [22]. High energy mills, with the capability to control the mill temperature like the one manufactured by Zoz GmbH, Germany [30], are increasingly being preferred by researchers.

2.6.2 Milling Container

The material for the vial or grinding vessel is important because it poses a risk of contamination. Most researchers on Ti-based alloys (including Ti-Mg and Ti-Al-Mg alloys) have used hardened steel [4, 5] or stainless steel vials [24, 31]. No contamination was observed in all these cases. Wilkes *et al* [23] used a titanium lined vial with hardened steel balls as the grinding media.

2.6.3 Milling Time

The milling time chosen depends on the type of mill, milling intensity, ball-to-powder weight ratio, milling temperature and need to prevent contamination [27]. Longer milling times produce finer particle sizes which facilitate

increased solubility [20]. However, the chances of contamination and formation of undesirable phases also increase with such milling times, and a balance needs to be found between increasing solubility without producing undesirable phase. Wilkes *et al* [23] and Sun *et al* [20] milled Ti-Mg alloys for 48h to achieve complete solid solutions of 9 wt % and 24 at% Mg in Ti respectively. Suryanarayana *et al* [19] observed that mechanical alloying started after 4 hours of milling. It is possible that differences observed by various researchers were caused by the type of mill used.

2.6.4 Grinding media

The choice of grinding media is important because it can be a source of contamination. For the milling of Ti-based alloys (including Ti-Mg and Ti-Al-Mg alloys), hardened steel and stainless steel balls have been used without any contamination being observed [1, 4-5, 20, 31].

Grinding media size is also important because it determines the amount of energy transferred to the powders per impact. Generally, large size of grinding media is useful since the larger weight of the balls will transfer more impact energy to the powder particles. Also, smaller diameter balls tend to produce amorphous phases [22].

2.6.5 Ball-to-Powder Weight ratio (BPR) and Charge Volume

Ball-to-powder weight ratio sometimes referred to as the charge ratio; determine the number of collisions per unit time and hence, the time to generate the required phases. Higher BPRs favour shorter milling times because the number of collisions per unit time is high and consequently more energy is transferred to the powder particles and alloying takes place faster. Ball-to-powder weight ratios can range from 1:1 to as high as 220:1 depending on the mill type [27]. Most researchers in the mechanical alloying of Ti-Mg and Ti-Al-Mg alloys have used ball-to-powder weight ratio ranging from 4:1 to 20:1 [4-5, 17, 22-25, 31].

2.6.6 Milling Atmosphere

The milling atmosphere is chosen so as to avoid powder contamination. The atmosphere chosen is determined by the reactivity of the powders being milled. Milling containers are usually evacuated or filled with an inert gas such as argon. Milling in the presence of nitrogen has been observed to minimize contamination, but this does not appear to be true for reactive powders such as titanium or its alloy powders [27]. Titanium based alloys (including Ti-Mg and Ti-Al-Mg alloys) have been synthesized under argon atmosphere and no contamination was recorded [2, 4-5, 19-20].

2.6.7 Process control Agent

True alloying among powder particles can occur only when a balance is maintained between cold welding and fracturing of particles and this is achieved by addition of a process control agent (PCA). PCAs are mostly organic compounds, which act as surface-active agents and are in the form of solids, liquid or gases. PCAs inhibit agglomeration while minimizing cold welding by adsorbing on the surfaces of powder particles. They are mainly used for ductile materials. A wide range of PCAs has been used in practice at a level of about 1-5 wt% of the total powder charge [27]. In any case, there has been no systematic study of the optimum amount of PCA reported in the literature, possibly because the optimum amount is system dependent.

The nature and amount of PCA used during milling determines the final powder particle size, powder yield (amount of free flowing powder recovered from the chamber) and final phases. An exponential decrease of the powder particle size has been reported with increasing PCA content for a given milling time [27]. PCAs affect the final phase formation by changing solid solubility levels and contamination levels [27].

The most important of the PCAs are mostly organic compounds and examples include stearic acid, hexane, methanol, and ethanol [27]. Stearic acid [20, 22, 24] has been mostly used as the PCA in the mechanical alloying

of Ti-based alloys. Occasionally, inorganic compounds e.g. NaCl [31] are used.

After milling, the loose powder can be consolidated into a shaped mass through application of external pressures, thereby giving the compact sufficient strength for subsequent handling [17].

2.7 Sintering

The sintering process involves conversion of loose powder particles into solid components through heating of the compacted powders at a chosen temperature under a controlled atmosphere for a given period of time. This process is well covered in many texts [e.g. 17] and the focus here will mainly be on the most important points pertaining to Ti alloys. The sections below cover some of the processes involved and factors which affect these processes.

Compacted metal powder green bodies are sintered /heat-treated at temperatures approximately half the absolute melting temperature to improve properties [17, 32]. Sintering can be performed simultaneously with compaction (hot pressing or hot isostatic pressing) [33]. The driving force for sintering is the reduction in the system surface energy. A characteristic feature of sintering is that it is temperature sensitive.

In many instances, sintering is accompanied by shrinkage; defined as the change in compact length from the green dimensions divided by the initial dimension. Similarly, densification parameter, Φ , is defined as the change in density from the green state divided by the maximum possible density change [17]. These parameters are used in evaluating progress achieved during sintering. Two types of sintering are possible: solid and liquid phase sintering. Solid state sintering occurs when powders remain in the solid state. Liquid phase sintering will be briefly covered as it is of much interest to this study.

2.7.1 Powder Compaction

Milled powders are consolidated into an initial solid mass, called a green compact by compaction. Loose powder particles, held in a container of desirable shape (die) are exposed to an external pressure applied by a press [17]. The effect of external pressure is shown in **Figure 2.3**: Excess void space decreases with increasing external pressure, resulting in a denser compact. As a result, the density of a green compact will depend on the pressure applied during compaction. Low *et al* [34] obtained an increase in the green density of compacts, with increasing pressure. Green density ranged from 63.4% to 79.9% of theoretical for pressures ranging from 200 to 600MPa. Green density is important because it affects sintering [17, 32].

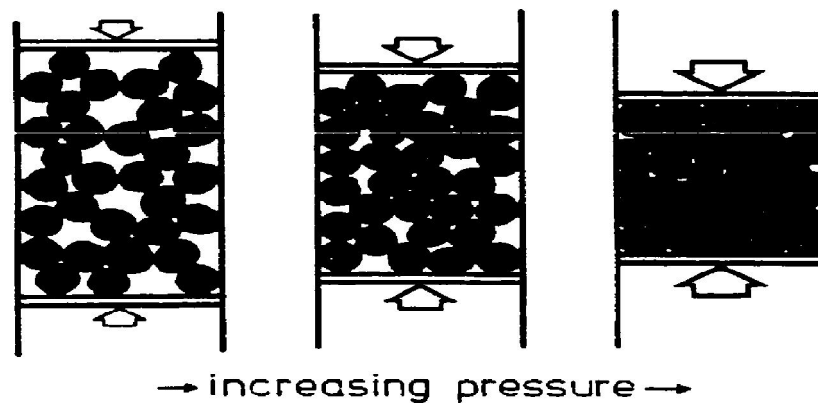


Figure 2.3: Stages of powder compaction [33].

The external pressure is applied on the powder, either at room or elevated temperature. Compaction that uses uniaxial pressurization of the powder in a die gives higher packing densities; but density gradients may result in the part since the powder does not respond uniformly to the applied pressure [17]. Thus, powders are sometimes isostatically pressed so that pressure is transmitted equally in all directions in processes called cold or hot isostatic pressing (CIP or HIP), depending on the temperature.

Higher pressures (1400MPa) are applied in CIP compared to HIP [35]. It would appear that HIP would not be well suited for Ti-Mg alloys because there is also the possibility of Mg evaporation especially if higher temperatures are used. Caetano *et al* [5] cold isostatically pressed mechanically alloyed Ti-10Al-5Mg powders at room temperature under a pressure of 330MPa. Nishiyama [36] used a pressure of 490MPa on Ti-Mg

alloys. Higher compression pressures lead to increased density and neck contact size, while reducing the rate of neck growth during sintering [17].

Cold compaction is aided by use of binders and lubricants which reduce friction between powder particles and also between the particles and the die and hence improve green compact properties and tooling life [33, 37]. For metal powders, stearates based on Al, Zn, Li, Mg or Ca, are commonly used as binders. Generally, the processing of titanium alloys presents a problem because of the need to eliminate oxidation or reaction with lubricants, hence high strength titanium alloys are sometimes consolidated without lubricants [38]. Binders and lubricants are eliminated, in a process called de-binding, before the green compacts are sintered.

Mechanical properties of the green compact, such as, the green density and green strength, are dependent on the mode of compaction, powder particle size distribution and powder lubrication. Defects generated in the compact during consolidation cannot be corrected by sintering. Thus optimum compact properties must be obtained during compaction.

2.7.1.1 Effect of Particle Characteristics on compaction

Powder characteristics affect the pressure-density-strength relationship in a powder compact [17]. Hard, stronger particles are difficult to compact since they need a higher compaction pressure. Generally the higher the particle hardness, the lower the green density at any given compaction pressure because both fracture and deformation are expected. Stronger particles can be obtained if the rate of work hardening is higher. As shown in **Figure 2.3**, particles deform during compaction.

Powder particle shape, size and size distribution affect packing efficiency during compaction and the resulting green compact properties. Dense packing is obtained from powders with optimized particle size distribution, i.e. selecting particles in such sizes and fractions that voids between larger particles are occupied by successively smaller particles. Fine particles press with difficulty because of a higher inter-particle friction. On the other hand, finer particles give more inter-particle bonding at a given green density and thereby promote higher green strength.

Particles with smooth surfaces tend to move easily relative to one another and produce compacts with low green strength. Irregularly shaped particles result in the optimal green strength because of mechanical interlocking [17,

39-40]. There does not appear to be any studies that have reported the effect of particle size on compaction for Ti-based powders.

2.7.1.2 Powder Lubrication

The effect of particle characteristics on green compact properties can be affected by the amount of binder/lubricant added to the powder. For example, excess lubricant lowers the green density and increases springback after the compaction pressure is removed. Since density is the primary determinant of strength, lower green density corresponds to lower green strength. Concentrations of lubricants usually range up to 2wt% [27], even though it is to be expected that the amount of lubricant is dependent on the powder characteristics.

2.7.2 De-Binding

Lubricants and/or binders, that are added to powders to improve the green compact properties after compaction by enhancing adhesion between the powder particles [33], are normally removed from the green compact prior to sintering by a burn-out step [37]. This is an important step because there is a general risk of the green compact weakening as the binder is removed, and also any residual products during de-binding impede the formation of good metallurgical bonds during sintering, hence affect the sinter properties. For

this reason, the burn-out is a generally low temperature process achieved at very low heating rates [41].

During debinding, the pore structure starts as a fully saturated body. As the binder softens, the green compact undergoes a strength loss; however, sintering generates strength in powder compact [42]. Binders can occupy up to 40% of green compact volume thus large shrinkages are observed after debinding; and this can result in shape distortion and by extension, poor mechanical and physical properties. Distortions in components due to poor debinding cannot be eliminated or corrected by sintering.

2.7.3 Stages of Solid State Sintering

The stages of sintering are as shown in **Figure 2.4**. The initial stage of solid state sintering is the rapid development of a neck between particles, driven by local curvature gradients [17]. The pore structure is open and connected and no change in dimensions occurs [33]. Material is transported on the surface of the particles. The intermediate stage involves the necks growing with time resulting in densification coupled with grain growth. Material transport occurs in bulk by either volume, grain boundary diffusion, or plastic and viscous flow. Although the pore structure becomes smooth, it remains interconnected.

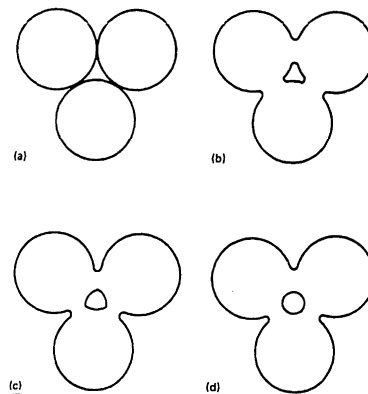


Figure 2.4: Three sphere sintering model: a) point contact, b) neck growth, c) pore closure and d) pore rounding [33].

The driving force is the interfacial energy associated with the pore structure including the surface and grain boundary energy. This is a weak force hence sintering rate is slow and sensitive to grain-pore morphology.

Grain growth can occur in the later portion of the intermediate stage. As a result, either pore motion or pore elimination can occur. The final stage of sintering is marked by the appearance of spherical, isolated pores. Material transport from the neck regions to the pore surface results in the pore becoming more rounded [33]. In this stage the kinetics are very slow since the driving force is strictly the elimination of the pore-solid interfacial area.

For this stage to occur solids must be transported into the pores and a means must exist by which the gas in the pores can escape to the surface. The

resultant effect is to decrease the volume of the sintering mass. Normally, a few isolated spherical pores remain in the material since complete elimination is impractical [17, 33]. It appears there are no published results of solid state sintering of Ti-Mg alloys.

2.7.4 Liquid Phase Sintering (LPS)

Liquid phase sintering involves a solid particulate and coexisting liquid during the sintering process [32, 42-44]. It occurs when the temperature is sufficiently high for one of the components in the green compact to partially melt. Mass transport is very rapid in the presence of a liquid phase, often over 100-times faster than in solids. A liquid phase provides both a capillary force and transport medium that leads to rapid consolidation and sintering [43]. The stages in liquid phase sintering are as shown in **Figure 2.5**.

LPS has been applied to various alloy systems including Ti-Mg [37] and Ti-Al-Mg [4, 5] alloy systems. In general, it is essential to restrict the amount of liquid phase in order to avoid impairing the shape of the part. LPS of Ti appears to be associated with swelling which has been attributed to the Kirkendall effect or rapid formation of intermetallics at the surface wetted by the liquid [34]. However, it (LPS) is still attractive to this study because it is associated with rapid compact densification at no external pressure.

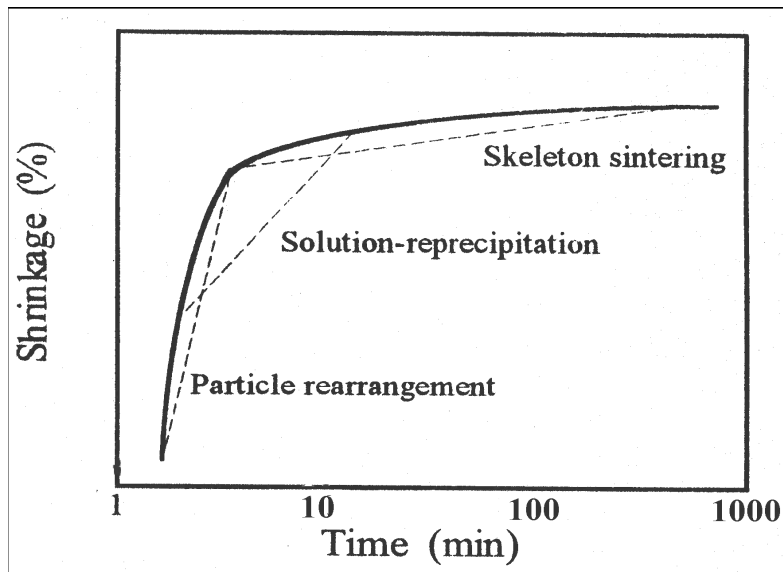


Figure 2.5: Schematic shrinkage curve for the three stages of LPS [44]

2.7.5 Sintering Parameters

The extent of sintering is affected by many variables which include temperature, time, atmosphere, heating rate, sintering pressure, alloy composition, green density and particle size. A greater control of these variables is required for optimum sintered properties. Generally, these parameters or variables are interdependent.

2.7.5.1 Temperature

Generally, powder particles sinter at high temperatures where mass transport by processes such as lattice diffusion becomes significant and densification is faster [17]. However at these same high temperatures, bulk material thermal

softening leads to significant strength loss. The sintering temperature appears to be dependent on the particle size. Fine particles (Fines) can be sintered at lower temperature. This is because the driving force, surface energy per unit volume, is greater [17]. Also, material transport for fine grain structures is better as a result of the higher defects that allow for faster diffusion [33, 35].

The optimum sintering temperature for any powder mixture is usually determined by differential thermal analysis (DTA) prior to the actual sintering in the furnace [2, 4-5, 19-20, 25, 45]. The analysis is carried out in an atmosphere and at a heating rate similar to that to be employed during sintering in the furnace.

2.7.5.2 Atmosphere

The sintering atmosphere selected is determined by the reactivity of the powders being sintered. Any powder surface contamination could hinder diffusion bonding and development of adequate properties. Various atmospheres are possible, including hydrogen, dissociated ammonia, inert gas, nitrogen based, vacuum and natural gas based. Because of the reactivity of Ti, only inert atmospheres of argon or vacuum have been used in the sintering of titanium based alloys [4, 5, 18, 34, 40]. It would appear that sintering under vacuum leads to better sintered densities than sintering in argon [18].

Sintering atmosphere can also affect the sintering mechanisms. Gas transport rates are high and major changes are possible in pore structure, grain shape, impurity content, and sintering kinetics through sintering atmosphere [32]. For example pore mobility and pore size are affected by vapour transport, presenting an opportunity to independently influence the grain growth and pore mobility rates via atmosphere and temperature control [17].

2.7.5.3 Sintering Furnaces

A sintering furnace provides the time-temperature control to the sintering cycle and the sintering atmosphere. It also provides for the removal of the lubricants and binders, and other subsequent heat treatments [17]. The furnace used in sintering metal powders depends on the quantity, material to be sintered, operating costs, type of atmosphere, and type of post-sintering cooling rate needed. Since this study aims to achieve full densification with pressureless sintering, a tube furnace is going to be used under argon atmosphere

2.7.5.4 Heating Rate

A high heating rate brings a green compact to the sintering temperature sooner. However, it may create large temperature gradients within the compact resulting in cracking, warping, or general shape distortion. For Ti-

based alloys, heating rates of between 10⁰C and 20⁰C/min have been employed [4-5, 34, 45].

Fast heating rates are applied for short times and densification is achieved. For smaller powder particles, low heating rates (hence longer heating time) may result in grain growth or coarsening. In such cases, novel heating mechanisms such as plasma heating, spark sintering, and plasma activated sintering, are applied [32].

2.7.5.5 Pressure

Pressure can be applied on compacts during sintering, usually at high temperatures to achieve full densification. Generally, full densification is difficult to achieve through pressureless sintering. Work on the sintering of Ti-Mg and Ti-Al-Mg has mostly been carried out at atmospheric pressure [4-5]. The specimens were observed to be porous after sintering. A value of 4.8% of open pores was determined by mercury porosimetry for a sample Ti-10Al-5Mg) sintered at 1100⁰C [5].

2.7.5.6 Alloy Composition

The reaction of a system to sintering depends on the relative solubilities of the species in one another, the relative surface energies and relative diffusion

rates [32]. The interfaces between phases aids vacancy creation while retarding grain growth. The compositional gradients require both time and temperature control to ensure homogenization. Mixed phase sintering is effective with fine or nanosized particles, since diffusion distances will be very small [17].

2.7.6 Compaction Effects on Sintering

Compaction affects the sintering behaviour of metal powders. Pressing a powder before sintering reduces the porosity while increasing the dislocation population in the powder. Thus there will be less shrinkage during sintering because of the reduced porosity. Higher dislocation density contributes to increased sintering rates [17]. Higher compaction pressures lead to increased density and neck contact size, while reducing the rate of neck growth during sintering. Also, shrinkage has been found to decrease with increasing compaction pressure [17].

2.7.7 Properties of Sintered Components

The properties of sintered components depend on the sintering conditions, pressed compact properties and powder characteristics. Common properties of concern are the compact hardness, strength, toughness/ductility, and density [17]. To obtain high mechanical properties in Ti alloys (in the as-

sintered condition), it is necessary to achieve higher sintered density while limiting the oxygen content below the upper limit value, above which oxygen degrades the mechanical properties [46].

Mechanical strengths (ranging from 264 to 667MPa) of sintered titanium powders are comparable to those of the wrought product of similar impurity content. However, ductility can be inferior to that of the wrought product [18]. Density decreases are expected after alloying Ti with low density alloying elements such as Mg and Al. There is also an expected improvement in the strength levels of Ti-Al-Mg alloys as a result of precipitation hardening [5].

2.7.8 Sintering of Ti-Mg and Ti-Al-Mg Alloys

A lot of work has been done on the synthesis of titanium based alloys, but very few researchers have worked on the sintering of Ti-Mg and Ti-Al-Mg alloys. Sintering data of Ti-Mg alloy powders has been very scarce compared to that of Ti-Al-Mg powders. Suryanarayana and Froes [19] annealed compacts of mechanically alloyed Ti-Mg powders at different temperatures. Magnesium losses were detected through EDS at higher annealing temperatures. They obtained very diffuse XRD patterns even on annealing up to 900K, due to heavy cold-welding experienced by the powders. Annealing at 1173K gave XRD patterns which could be indexed on the basis of elemental Ti and Mg indicating that equilibrium had been attained at this

stage. No information was given regarding the properties of these annealed compacts.

Nishiyama [36] sintered Ti-Mg compacts at temperatures ranging from 610⁰C to 700⁰C for 10 to 60 minutes, under pressures of 60MPa. A liquid phase was formed for temperatures from 657⁰C since these temperatures were above the melting temperature of the lower melting point phase, magnesium (m.pt = 650⁰C). Sintering was carried out at atmospheric pressure. Lu *et al* [47] worked on the sintering of Mg based Mg-Ti, Mg-Ti-Al, Mg-B and Mg-Ti-Al-B alloys. The compacts were sintered at 450⁰C in an argon atmosphere for two hours in a tube furnace.

Caetano and Trindade [5] sintered a Ti-10Al-5Mg (wt %) alloy at 900 and 1100⁰C in vacuum for 12 hours at a heating rate of 0.25⁰C/s. The scanning electron microscope (SEM) images showed lower porosity on samples sintered at 1100⁰C. At these high temperatures, Mg reacted with oxygen to form MgO while Ti formed α_2 -Ti₃Al. Values of 11 and 188GPa were obtained for the hardness and Young's Modulus respectively, for samples sintered at 1100⁰C which confirms the strength improvement of the Ti-based alloy by the α_2 -Ti₃Al+MgO reinforcements.

Trindade *et al* [4] sintered Ti-10Mg-5Al (wt %) alloy powder compacts at 600 and 900°C. Hardness tests for the samples confirmed strength improvement of the Ti-based alloy due to the MgO reinforcement. An average hardness value of about 4.5 GPa was obtained. At a temperature of 833K, Ding *et al* [48] applied Spark Plasma Sintering (SPS) on Ti-Mg alloy samples and densification was enhanced. The powders were mechanically mixed and blended for enough time to make them homogeneous. In these studies, no comparisons were made between the theoretical density and the sintered density attained. Most researchers [4-5] carry out pressureless sintering under an inert atmosphere or vacuum. Components produced under such conditions are expected to be porous.

2.8 Motivation for this project

A lot of work has been done on the production of Ti-based alloys. But most of the work did not involve alloying Ti with Mg because Mg has a very low solubility in Ti under equilibrium conditions. Only a few researchers have produced Ti-Mg based alloys through both mechanical alloying and sintering. Most researchers concentrated on mechanical alloying only. Thus literature on the sintering of Ti-Mg based alloys is very limited. Parameters which affect sintering such powder particle size and compaction pressure have not been investigated. Effect of these parameters on green density will be examined.

Sintering has also been carried out at higher temperatures (up to 1100°C) but this resulted in high Mg losses through evaporation. Therefore, relatively lower temperatures will be used (between 630 and 900°C). Mg is expected to be retained in the solid solution at these temperatures. Not much has been reported on the sintered body properties. This project will report on sintered properties such as sintered density, hardness and phase constituency. Chemical composition will be determined using EDS. Effects of compaction pressure and powder particle size on sintered properties will be investigated.

CHAPTER THREE

EXPERIMENTAL PROCEDURE

3.1 Materials

The materials used in this project were powders provided by CSIR, Pretoria. The nominal chemical compositions of the powders were: Ti-10Mg (wt. %); Ti-20Mg (wt. %) and Ti6Al4Mg (wt. %). The powders had been milled for various durations between 16 and 32 hours to obtain Ti-alloy powders, as shown in **Figures 3.1 – 3.3**, which summarise the information that was provided by CSIR.

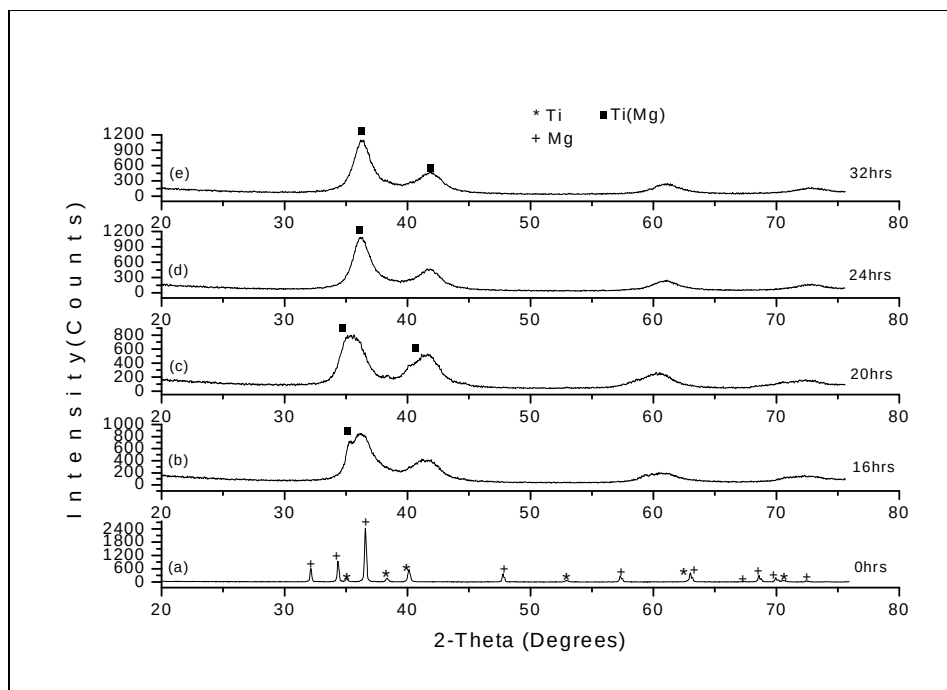


Figure 3.1: XRD spectra of milled Ti-10Mg powders.

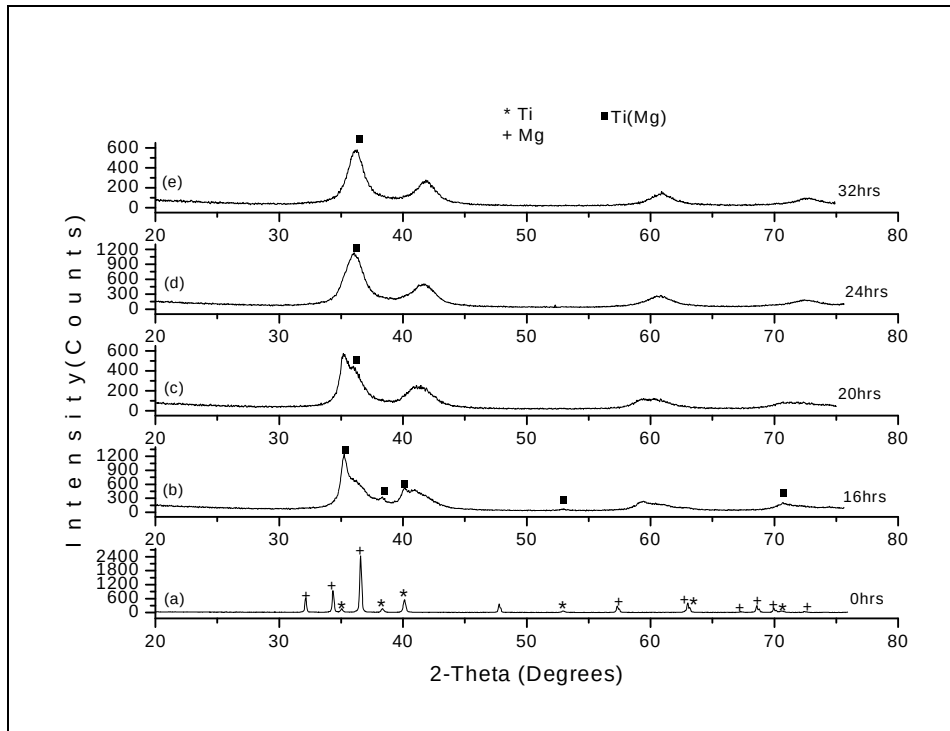


Figure 3.2: XRD spectra of milled Ti-20Mg powders

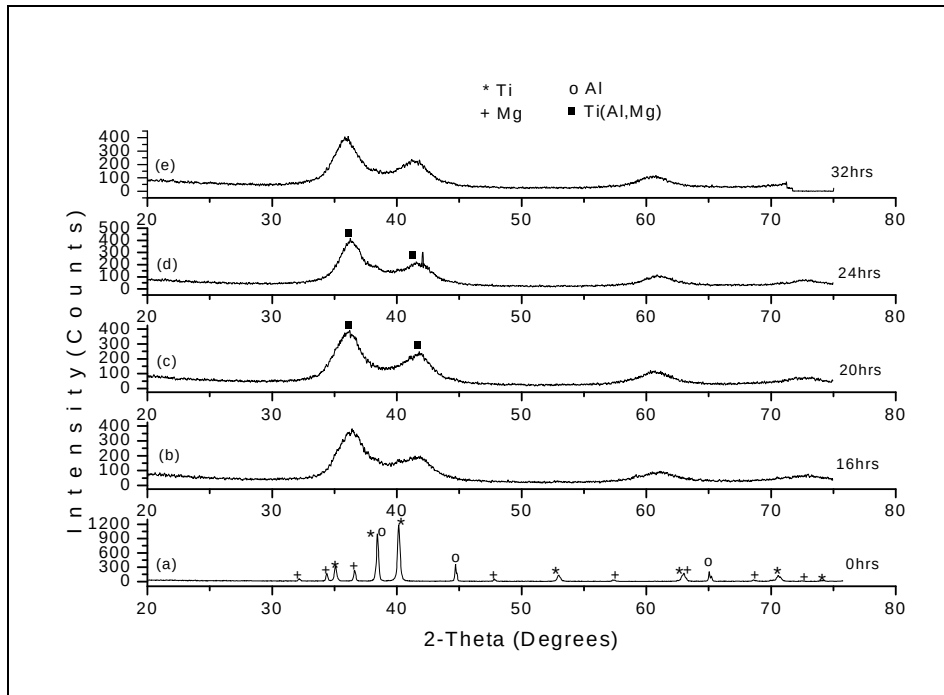


Figure 3.3: XRD spectra of milled Ti-6Al-4Mg powders

Broadening of XRD peaks observed in milled samples compared with the unmilled starting materials (**Figures 3.1 to 3.3**) is attributed to an accumulation of micro-strains/ lattice strains as a result of the milling action coupled with a crystallize size effect. Energy Dispersive Spectroscopy analysis on randomly selected particles showed that the powders could be considered chemically homogenous. Compositional difference between the starting powders and the milled powders was negligible as shown by the amount of Mg and Al in the milled powders (**Table 1 – 3**). Mg contents of 8.84 - 9.64 wt% and 18.87 – 20.12 were obtained in Ti-10Mg and Ti-20Mg powders respectively.

Table 1: Mg contents of milled Ti-10Mg powders as determined by EDS

Alloy powders	Mg Content (wt. %)
Ti-10Mg 16 hours	9.64
Ti-10Mg 20 hours	8.96
Ti-10Mg 24 hours	9.23
Ti-10Mg 32 hours	8.84

Table 2: Mg contents of milled Ti-20Mg powders as determined by EDS

Alloy powders	Mg Content (wt. %)
Ti-20Mg 16 hours	18.87
Ti-20Mg 20 hours	19.54
Ti-20Mg 24 hours	19.85
Ti-20Mg 32 hours	20.12

Table 3: Al and Mg contents of milled Ti-6Al-4Mg powders as determined by EDS

Specimen	Al Content (wt. %)	Mg content (wt. %)
Ti-6Al-4Mg 16 hours	5.64	3.86
Ti-6Al-4Mg 20 hours	5.46	3.98
Ti-6Al-4Mg 24 hours	5.86	3.87
Ti-6Al-4Mg 32 hours	5.98	4.02

3.2 Particle Size Analysis

Particle size distributions (d_{10} , d_{50} and d_{90}) were determined by laser scattering in the wet cell of a Malvern mastersizer 2000 particle size analyser. The powder particles were dispersed in water and a few drops of acetone which was added as a dispersant. The wet unit was equipped with an ultrasonics agitator and a pump to circulate the powder suspension through the sample cell. The raw data from the particle size analyser was also used to plot cumulative-volume percent passing graphs so that comparisons of particle size distributions (for different milling times) could be made.

3.3 Powder Compaction and Green Density

Cylindrical compacts of approximately 13.86 mm in diameter and 4.66 mm in height were prepared from the mechanically alloyed powders using a uniaxial, single action laboratory press (**Figure 3.4**). The prepared compacts had an average mass of 1.9 g. On average, four (4) specimens were made for each composition and milling time. Compaction pressures of 27 MPa and 33 MPa were applied. Paraffin wax was used as a die-wall lubricant to facilitate pressing and ejection. Consolidation was carried out at room temperature to avoid possible loss of Mg through evaporation at high temperatures.



Figure 3.4: Laboratory press. (Above right is the picture of the pressure gauge)

The green density measurement of each compact was obtained by using the dry weight and the average dimensions (i.e. the diameter and thickness) of each sample measured using vernier callipers. The average of the measurements was used to calculate the volume. The density of each sample was determined using the following equations:

$$V = \pi r^2 h \quad (3.2)$$

$$D = M/V \quad (3.3)$$

Where V is the volume in cm^3 , h is the height of sample in cm , r is the radius of sample in cm , M is the weight in g and D is the density in g/cm^3 . Plots of green density-compaction pressure-milling time were made.

3.4 Sintering

Sintering of the compacted powders was carried out in a tube furnace (**Figure 3.5**) under argon and under atmospheric pressure. The samples (four at a time) were loaded into a yttria-coated alumina boat and placed at the centre of the tube. After charging, the furnace was flushed out to remove residual air by flowing argon for about 15 minutes prior to commencement of the heating cycle. A heating rate of $10^{\circ}C/minute$ was applied throughout. Sintering was done at temperatures of $630^{\circ}C$, $750^{\circ}C$ (for Ti-Mg powders) and $900^{\circ}C$ (for Ti-6Al-4Mg) (**Table 4**). However, no appreciable sintering took place at $630^{\circ}C$, thus only results for $750^{\circ}C$ and $900^{\circ}C$ are reported.

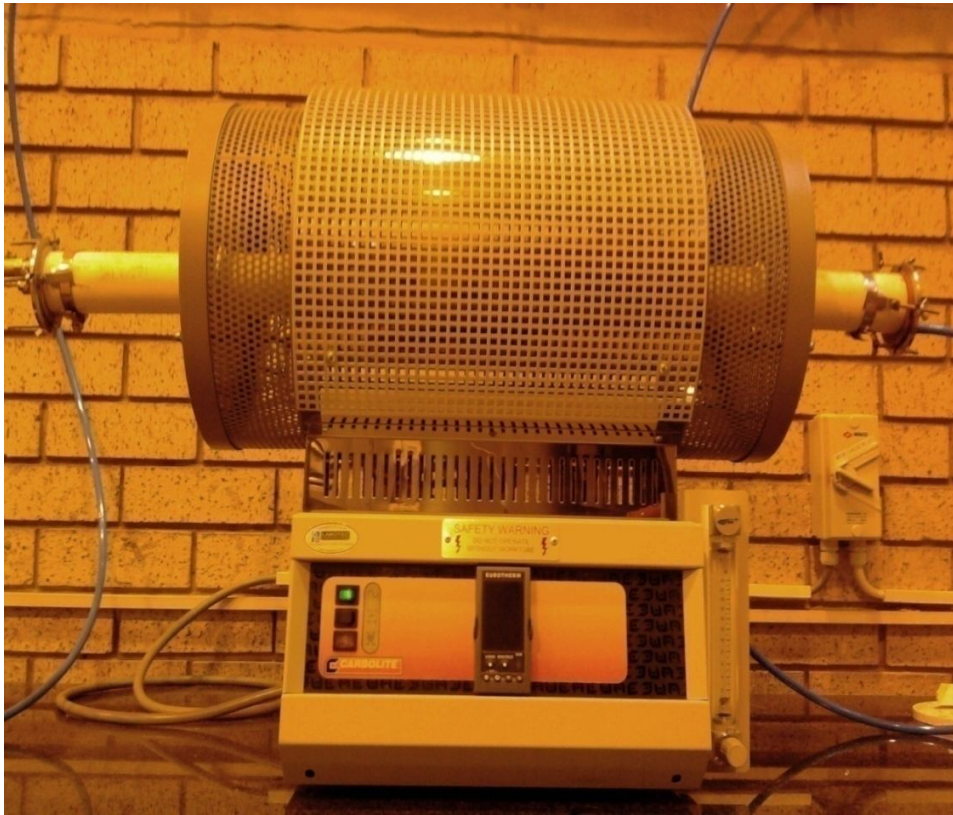


Figure 3.5: Picture of the tube furnace

Table 4: Sintering temperature / time combinations

Alloy	Sintering Temperature(°C),Time (hours)
Ti-10Mg	630 °C, 10hours and 750°C, 5hours
Ti-20Mg	630 ⁰ C, 10hours and 750°C, 5hours
Ti-6Al-4Mg	900 ⁰ C, 10hours

The heating cycle during sintering included a debinding step, which was done at 250⁰C for 1.5 hours. At the end of the sintering cycle, specimens were allowed to furnace cool to room temperature.

3.5 Characterization of sintered parts

3.5.1 Sintered Density

Archimedes' principle (which states that the volume of an object is the difference in wet weight of the object in air and that of the object's weight suspended in water) was used to determine the sintered densities of the specimens. The densities were calculated using **equation (3.4)**:

$$r_s = (m_s \cdot r_w) / (m_s - m_w) \quad (3.4)$$

Where r_s = sintered density (g/cm^3), r_w = density of water at room temperature ($\sim 1 g/cm^3$), m_s = mass of the sintered body in air (g) and m_w = mass of sintered body in water (g).

3.5.2 Microscopy

Optical micrographs were obtained using a Zeiss Axiotech microscope mounted with a camera. Micrographs were taken at magnifications of 200 and 1000. Images were also acquired using a JEOL JSM-6510 Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray Spectroscopy (EDS). This scanning electron microscope was operated at accelerating voltages of between 10kV and 20kV. Working distances of between 9 and 12 mm were applied. Images were acquired at magnifications ranging from 200

to 30000, using both back-scattered electron (BSE) and secondary electron (SE) modes. Elemental composition was determined using Energy Dispersive Spectroscopy (EDS).

3.5.3 Hardness Tests

Hardness tests were performed using a digital Future Tech Vickers Hardness tester FV-700 (**Figure 3.3**). A test load of 5kgf was applied for a dwell time of 10 seconds.



Figure 3.6: Future Tech Vickers Hardness Tester FV-700

Ten (10) readings were taken from the cross-sections of the samples and the average value calculated. The values were expressed in Vickers Hardness (HV).

3.5.4 Phase composition

Phases present were determined (as in **3.2.1**) by means of a Phillips X-ray Diffractometer (Type PW 1710) equipped with Phillips Analytical X'pert Highscore software. A monochromatic $Cu-K\alpha$ radiation at 40kV and 20mA was used over 2Theta (2θ) angles between 20 and 80°. A step scan time of 2.5 seconds was applied.

3.6 Sample Preparation

Specimens for microstructural analysis and hardness measurements were prepared using a set of ATM equipment. The sintered samples were cross-sectioned using an ATM Sample cutter. Cross-sections were mounted in conductive Bakelite resin using an ATM Opal450 mounting machine, followed by wet grinding on silicon carbide (SiC) abrasive papers. The grades of grinding paper used ranged from 320 to 4000. Polishing was performed on rotating discs covered with soft cloth impregnated with 1 micron diamond paste. ATM Saphir550 equipment was used for grinding and polishing. After polishing, surfaces were cleaned in alcohol and dried using a blow dryer.

CHAPTER FOUR

RESULTS AND DISCUSSION

This Chapter presents the results of the various characterization techniques presented in Chapter 3. The characteristics of the powders will be presented first, followed by those of the sintered components.

4.1 Powder Particle Size Analysis

4.1.1 Ti-10Mg powders

Figure 4.1 shows the cumulative particle size distribution curves for the Ti-10Mg unmilled and milled alloy powders while **Figures 4.2-4.4** show the normal distribution curves for the unmilled and the powders milled 16 and 32 hours. Similar distribution curves were obtained for the other milling durations (**Appendix A**). The powder particle size distributions are given in **Table 5**. Kinks observed on **Figure 4.1** are due to the bi-modal particle size distribution (Refer to **Figures 4.3** and **4.4**).

The unmilled powder mixtures were characterised by a narrower particle size distribution compared to the milled powders (**Figure 4.1**). The unmilled

powder mixture had a mono-disperse particle size distribution while the milled powders had a bi-modal particle size distribution (**Figures 4.2 – 4.4**). Milling led to a reduction of the mean particle, and the particle size distribution curves shifted to the left compared to before milling (**Figure 4.1**). The average particle size decreased, moving from 53 μm (for the unmilled powder mixture) to 9.5 μm after 16 hours of milling. A gradual increase in the mean particle size was then observed on continued milling to just over 12 μm after 32 hours (**Table 5**). This increase is likely due to cold-welding dominating fracturing.

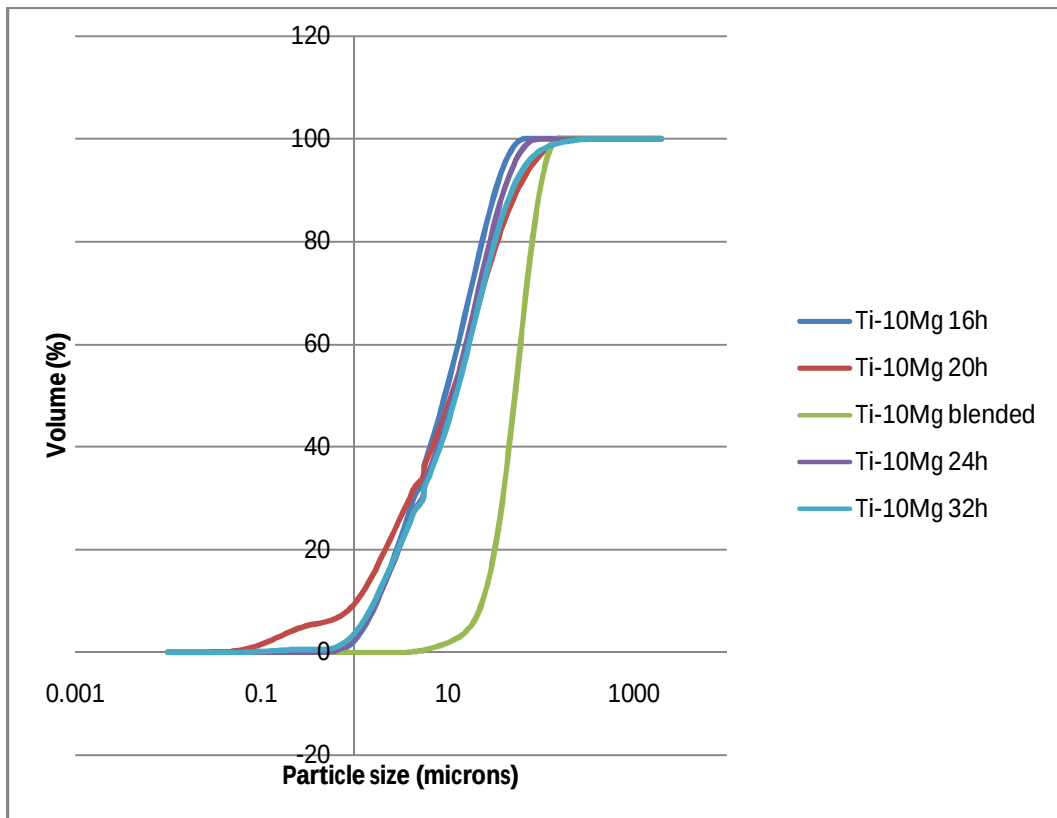


Figure 4.1: Cumulative particle size distribution curves for unmilled and milled Ti-10Mg powders.

While the cumulative distribution curves after milling were largely similar, the curve for the powder milled for 20 hours was initially raised more than for the other milled powders. This suggests that milling for 20 hours generated a higher amount of fine particles (i.e. fines) than the other milling durations.

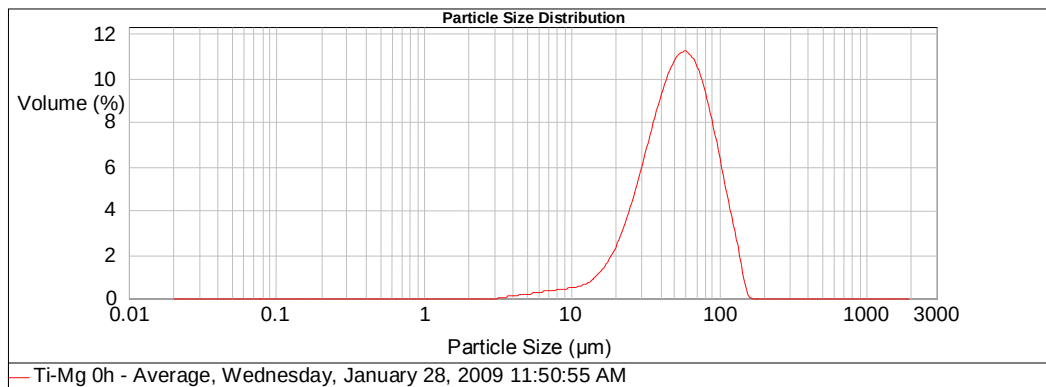


Figure 4.2: Normal distribution curve for the as-blended Ti-10Mg powders

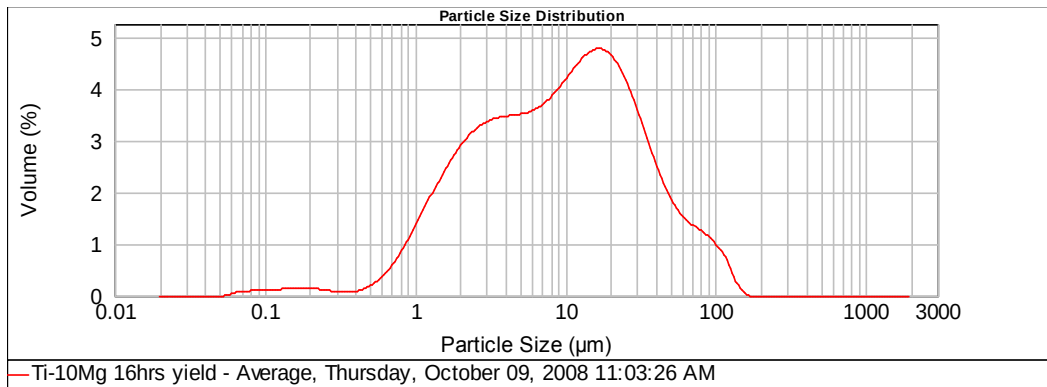


Figure 4.3: Normal distribution curve for Ti-10Mg powders milled for 16h.

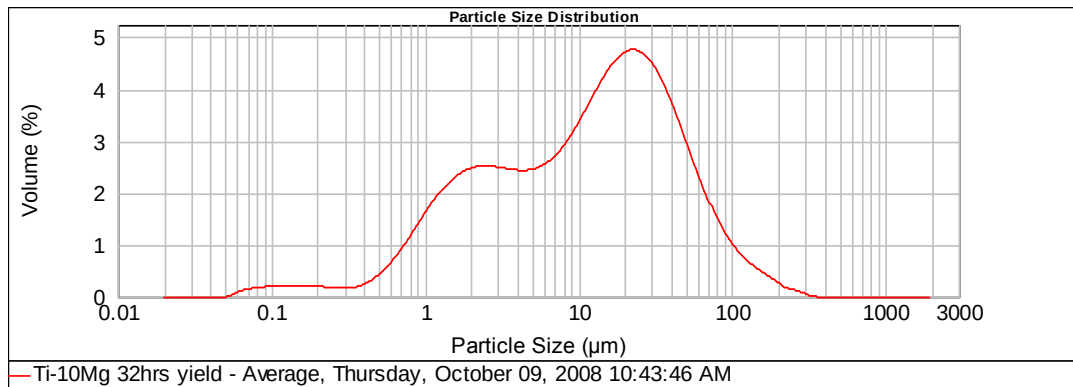


Figure 4.4: Normal distribution curve for Ti-10Mg powders milled for 32h

Table 5: Particle size distribution of Ti-10Mg powders

Powders	$d_{10}(\mu\text{m})$	$d_{50}(\mu\text{m})$	$d_{90}(\mu\text{m})$
Ti-10Mg 0hrs	24.052	53.394	104.325
Ti-10Mg 16hrs	1.573	9.609	42.763
Ti-10Mg 20hrs	1.106	10.193	44.484
Ti-10Mg 24hrs	1.373	10.970	46.894
Ti-10Mg 32hrs	1.274	12.727	54.734

4.1.2 Ti-20Mg powders

Figure 4.5 shows the cumulative particle size distribution curves for the Ti-20Mg powders. The particle size distributions (**Figure 4.5** and **Table 6**) and the effect of milling on powder particle size were similar to the observations made for Ti-10Mg (**Figure 4.1** and **Table 5**). A comparison between Ti-10Mg

and Ti-20Mg milled powders (**Tables 5 and 6**) shows that the Ti-20Mg powders were relatively coarser than those of Ti-10Mg. Thus the amount of Mg in the starting materials might be affecting the final or milled powder particle sizes.

The particle sizes of the milled powders were finer than the unmilled powder (**Table 6**). As for Ti-10Mg powders, the finest particles were obtained from powders milled for 20 hours. An average particle size (d_{50}) of 11.7 μm was measured for powders milled for 20 hours, which rose to about 15 μm after 24 hours of milling. This increase in particle size can be attributed to cold-welding. After 32 hours, d_{50} decreased to 14 μm , probably as a result of an increase in the fracturing tendency of the powder particles.

The normal distribution curves for Ti-20Mg powders showed a single bell shape (**Figure 4.6 and 4.7**) as opposed to the bi-modal particle size distribution observed for Ti-10Mg powders (**Figures 4.3 and 4.4**).

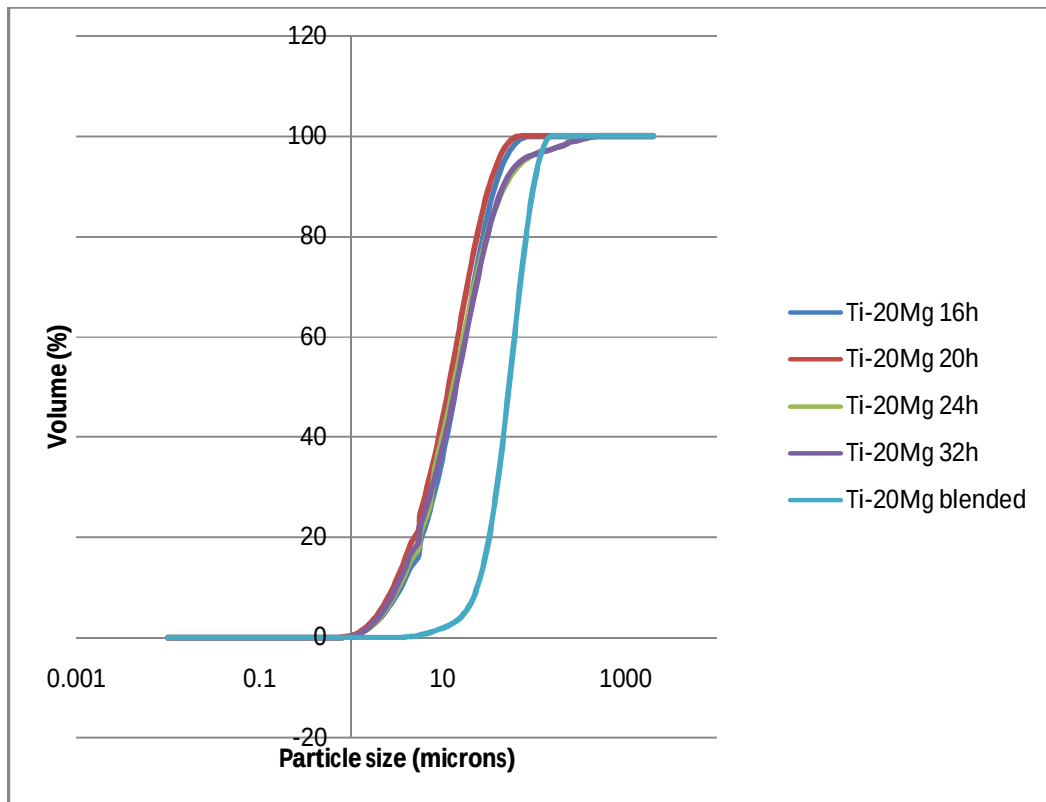


Figure 4.5: Cumulative particle size distribution curves for Ti-20Mg powders

Table 6: Particle size distribution of Ti-20Mg powders

Powders	$d_{10}(\mu\text{m})$	$d_{50}(\mu\text{m})$	$d_{90}(\mu\text{m})$
Ti-20Mg 0hrs	25.154	46.214	106.852
Ti-20Mg 16hrs	3.028	13.527	39.054
Ti-20Mg 20hrs	2.847	11.573	31.897
Ti-20Mg 24hrs	2.958	12.774	36.101
Ti-20Mg 32hrs	2.957	13.361	41.210

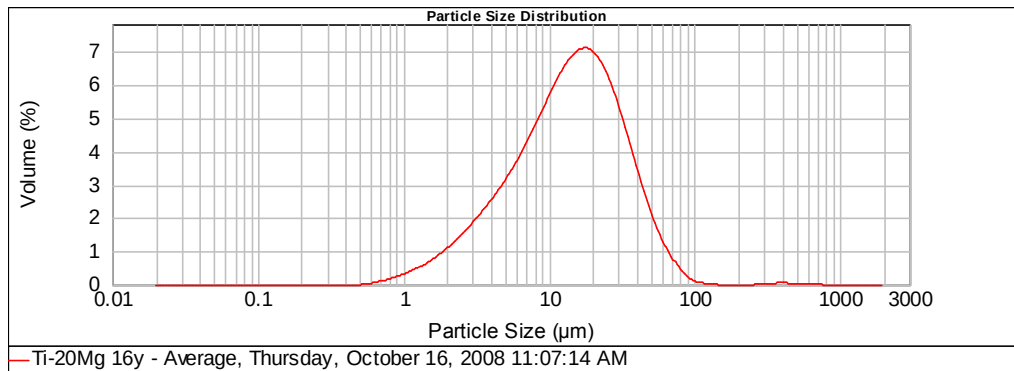


Figure 4.6: Normal distribution curve for Ti-20Mg powders milled for 16h

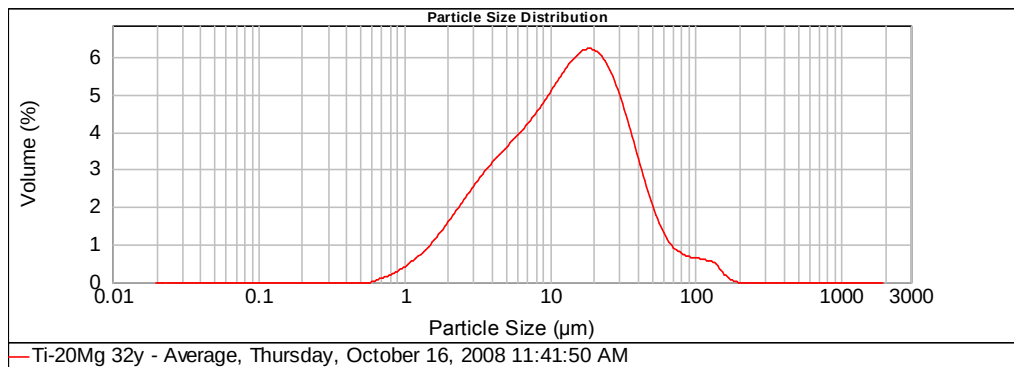


Figure 4.7: Normal distribution curve for Ti-20Mg powders milled for 32h

4.1.3 Ti-6Al-4Mg powders

Figure 4.8 shows the cumulative particle size distribution curves of blended and milled Ti-6Al-4Mg powders. Kinks observed on the graphs of the milled powders are due to the bi-modal particle size distribution. **Table 7** shows the particle size distributions. The effect of milling on powder particle size and size distribution was similar to that observed on Ti-Mg powders. A

comparison of the mean particle sizes of all the milled powders (**Tables 5 – 7**) indicates that the finest particles were obtained in Ti-6Al-4Mg powders.

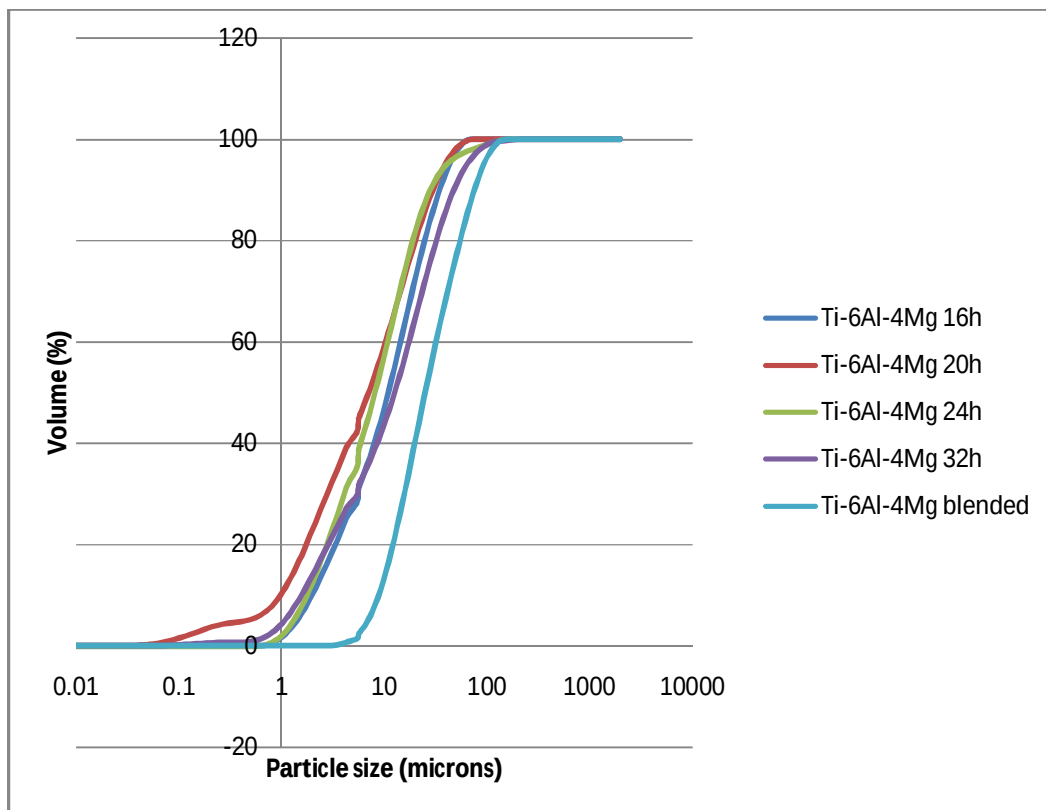


Figure 4.8: Cumulative particle size distribution curves for Ti-6Al-4Mg powders.

The finest particles of the milled Ti-6Al-4Mg powders were measured in powders milled for 20 hours. This was also observed in the other alloy powders. It would appear therefore that milling for 20 hours produced the finest particle sizes irrespective of the alloy composition. An average particle size of just over 7 μm was obtained for powders milled for 20 hours. The

mean particle sizes increased on further milling: The average particle size rose to almost 13 μm after 32 hours of milling indicating that cold-welding dominated fracturing after 24 and 32 hours, resulting in an increase in the particle sizes.

Table 7: Particle size distribution of Ti-6Al-4Mg powders

Powders	d₁₀(μm)	d₅₀(μm)	d₉₀(μm)
Ti-6Al-4Mg 0hrs	8.909	25.228	76.048
Ti-6Al-4Mg 16hrs	1.627	10.291	32.216
Ti-6Al-4Mg 20hrs	0.674	6.571	27.493
Ti-6Al-4Mg 24hrs	1.351	8.803	28.636
Ti-6Al-4Mg 32hrs	1.103	10.281	40.114

The normal particle size distribution curves were similar to those of Ti-10Mg powders. They reveal a bi-modal particle size distribution (**Figure 4.9 and 4.10**).

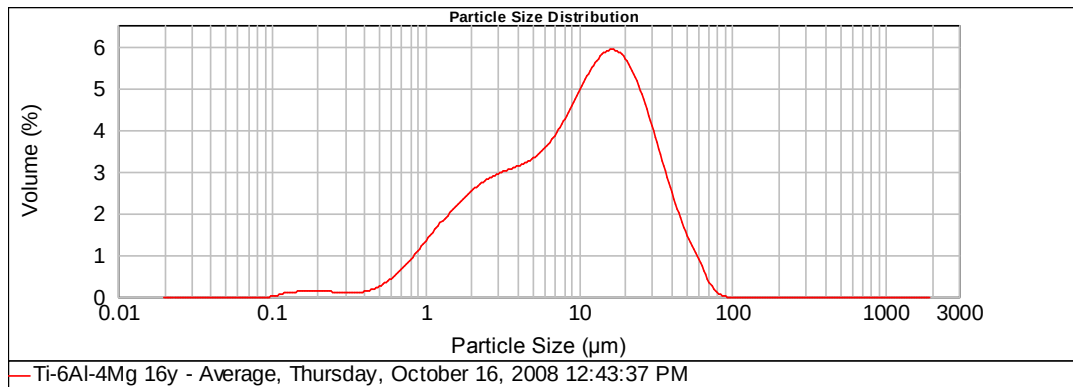


Figure 4.9 shows the normal distribution curve for Ti-6Al-4Mg powders milled for 16h

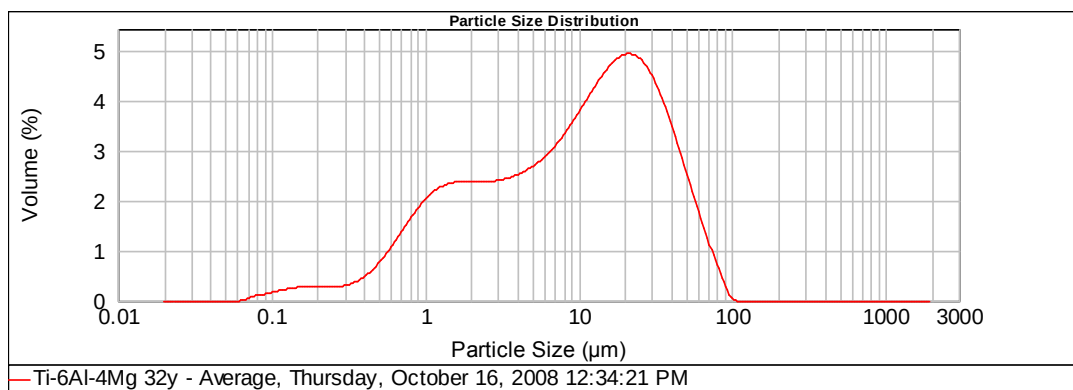


Figure 4.10 shows the normal distribution curve for Ti-6Al-4Mg powders milled for 32h

Particle size and size distribution are important for pressing. Very fine particles have a higher surface area that generates a higher inter-particle friction that reduces the effective force on the individual particles. This results in lower green densities. Thus relatively lower green densities might be

expected on Ti-6Al-4Mg powders and also on Ti-10Mg and Ti-20Mg powders milled for 20 hours.

4.2 Green density and Compaction Effects

Figures 4.11 - 4.13 (and **Appendix B**) show the variation of green density with milling time for the powders pressed in this study. As indicated earlier, the green densities were obtained from the masses and volumes of the green compacts, where volumes were obtained from the physical dimensions of the green compacts (Cf Section 3.3). Most of the compacts had high edge retention and were sufficiently strong for further handling.

The green densities generally decreased with increased milling time regardless of the compaction pressure used. A change in compaction pressure caused a significant change in the green densities, as can be seen from the fact that the error bars are not overlapping. Powders milled for 20 hours had the lowest green densities. This has been attributed to the finer powder particle sizes which make compaction difficult. Very fine particles have relatively higher surface area generating higher inter-particle friction that results in a decrease of the effective force.

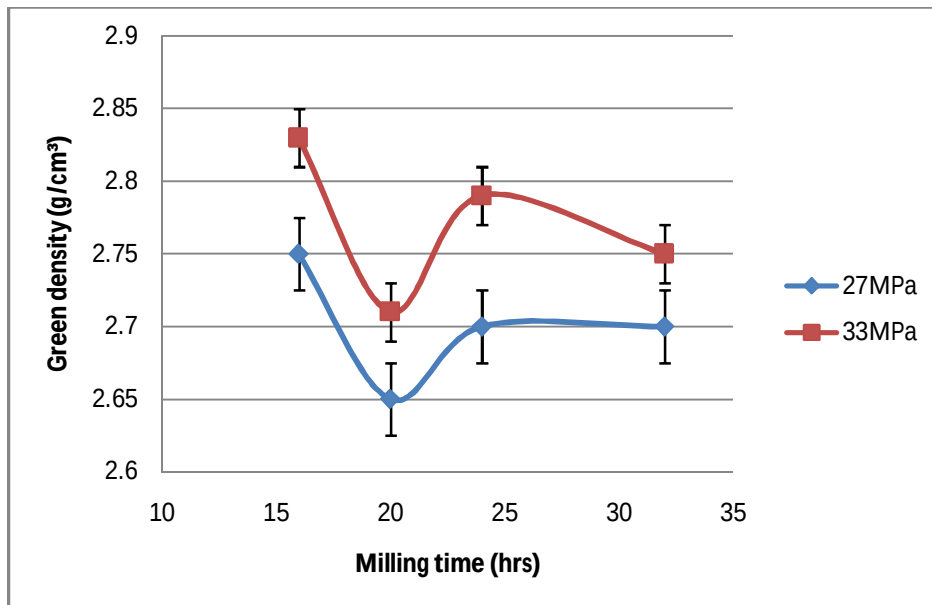


Figure 4.11: Green density- milling time graphs for Ti-10Mg powders pressed at 27 and 33 MPa.

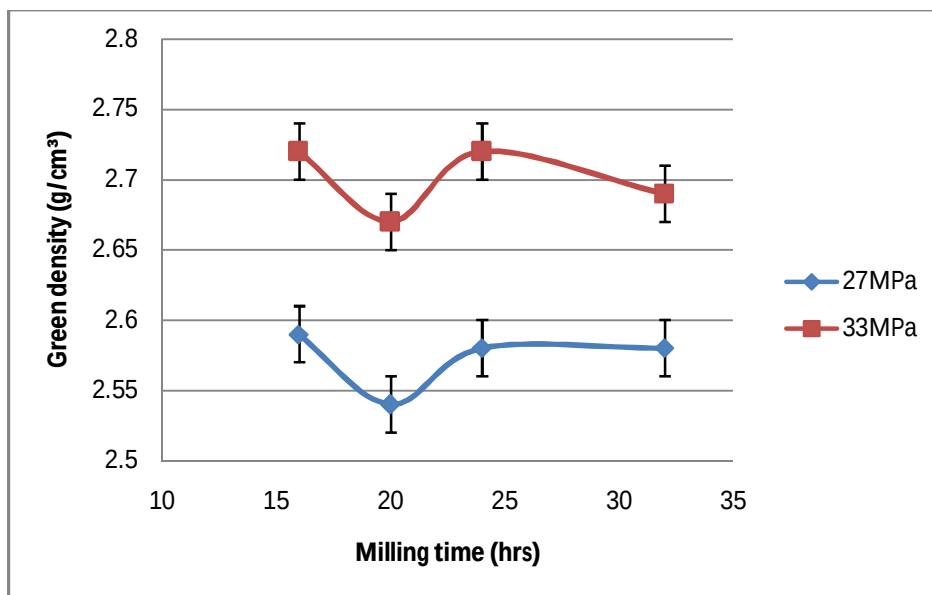


Figure 4.12: Green density- milling time graphs for Ti-20Mg powders pressed at 27 and 33 MPa

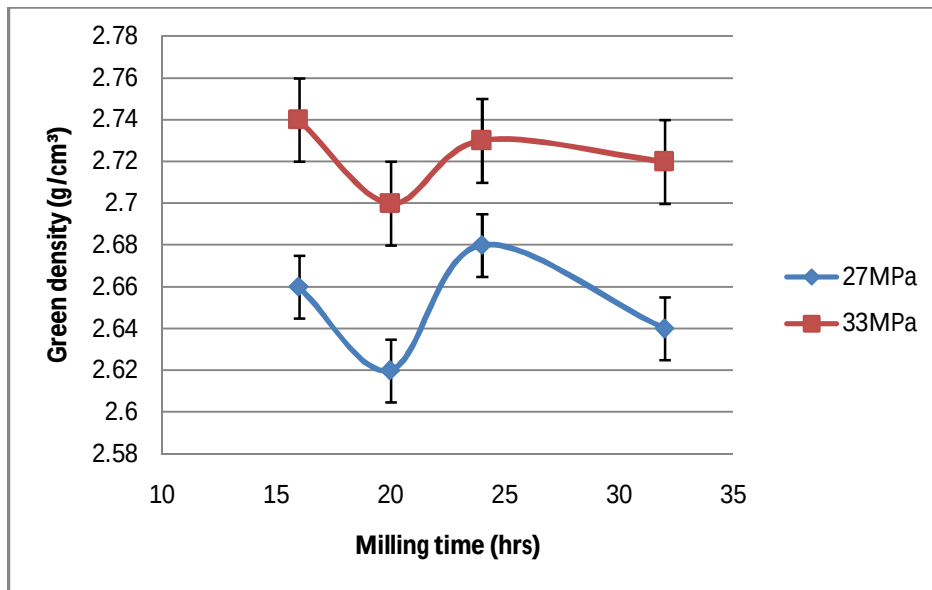


Figure 4.13: Green density-milling time graphs for Ti-6Al-4Mg powders pressed at 27 and 33 MPa

As expected, compacting at 33 MPa achieved higher green densities compared to compacting at 27 MPa. Increase in green density with pressure is attributed to increased particle-particle contact as a result of particles rearranging and sliding over each other. Higher pressures also encourage deformation and cold-welding between particles, hence increase in density.

The variations of the approximate percent theoretical density of the green densities are shown in **Figures 4.14** and **4.15**. The approximate theoretical densities of the three (3) alloys were calculated from the rule of mixtures (**Appendix C**) and are given in **Table 8**. These values are only an approximation because the lattice parameters of crystal structure of the alloys

formed in the powders have not been determined. As expected, incorporation of Mg in Ti favours an overall density decrease because of its low density.

Table 8: Relative density of powder mixtures

Powder	Density from rule of mixtures (in g/cm^3)
Ti-10Mg	3.89
Ti-20Mg	3.42
Ti-6Al-4Mg	4.08

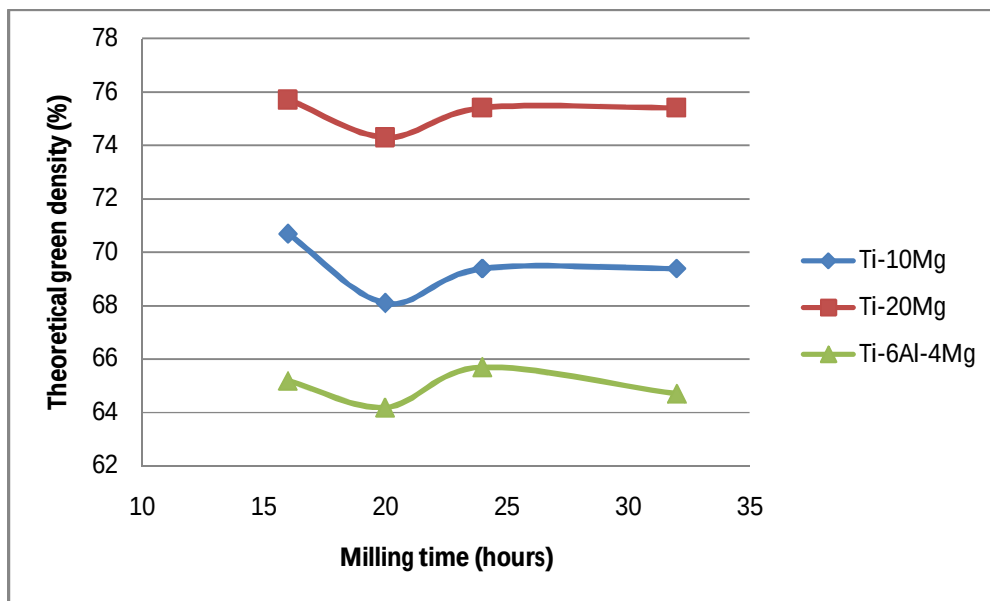


Figure 4.14: Percent theoretical density-milling time graphs for Ti-10Mg, Ti-20Mg and Ti-6Al-4Mg powders pressed at 27 MPa

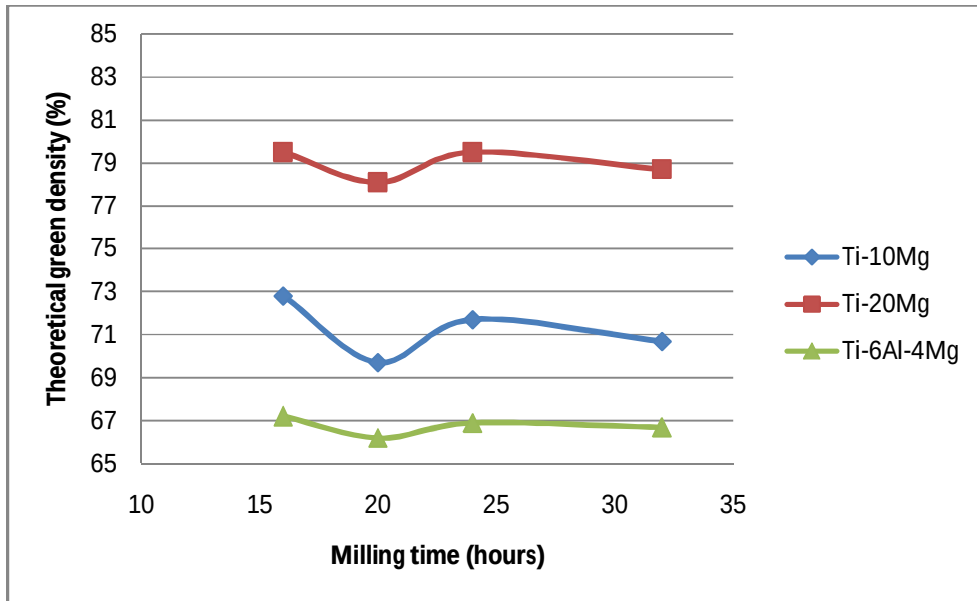


Figure 4.15: Percent theoretical density-milling time graphs for Ti-10Mg, Ti-20Mg and Ti-6Al-4Mg powders pressed at 33 MPa

Ti-6Al-4Mg powders pressed at 27 MPa had an average theoretical density of 65% compared to 67% for those compressed at 33 MPa. Ti-10Mg powders averaged between 69 and 72% while Ti-20Mg powders averaged between 75% and 79%, respectively. The variation of percent theoretical densities can be attributed to the changing fineness of the average particle size. The average particle sizes increase in the order: Ti-6Al-4Mg < Ti-10Mg < Ti-20Mg (Cf Tables 5 – 7). The larger powder particles in Ti-20Mg powders could be a contributing factor to the higher percent theoretical densities obtained here because coarser particles are easier to press than finer ones [17].

4.3 Sintered Properties

4.3.1 X-Ray Diffraction for the Sintered Compacts

Sintering green compacts of Ti-10Mg and Ti-20Mg at 630°C for 10 hours did not produce appreciable sintering, suggesting that the temperature was too low. All the Ti-10Mg and Ti-20Mg samples were therefore sintered at 750°C. Ti-6Al-4Mg specimens sintered well at 900°C.

4.3.1.1 XRD for Sintered Ti-10Mg compacts

The XRD patterns for Ti-10Mg compacts pressed at 27 MPa were almost similar (**Figure 4.16**). The only noticeably different pattern was for the specimen obtained from powder milled for 20 hours, which had fewer peaks: however, the 2Theta positions of these peaks were not different from those of the other specimens.

Comparing **Figure 4.16** to **Figure 3.1**, the XRD patterns of the milled powders, shows that the XRD peaks at around 36° (2Theta) and 41° (2Theta) in **Figure 4.16** have been retained from the milled powders, indicating that the Ti(Mg) solid solutions formed in the powders have been retained after sintering. This is in line with previous work [51]. No Mg peaks were observed on the XRD patterns of the sintered components (**Figure 4.16**). However,

new Ti peaks formed, indicating that the solid solution- formed in the powders had undergone some decomposition. It was observed that peaks became sharper after sintering. This can be attributed to an increase in grain size due to coarsening and also Mg coming out of solution.

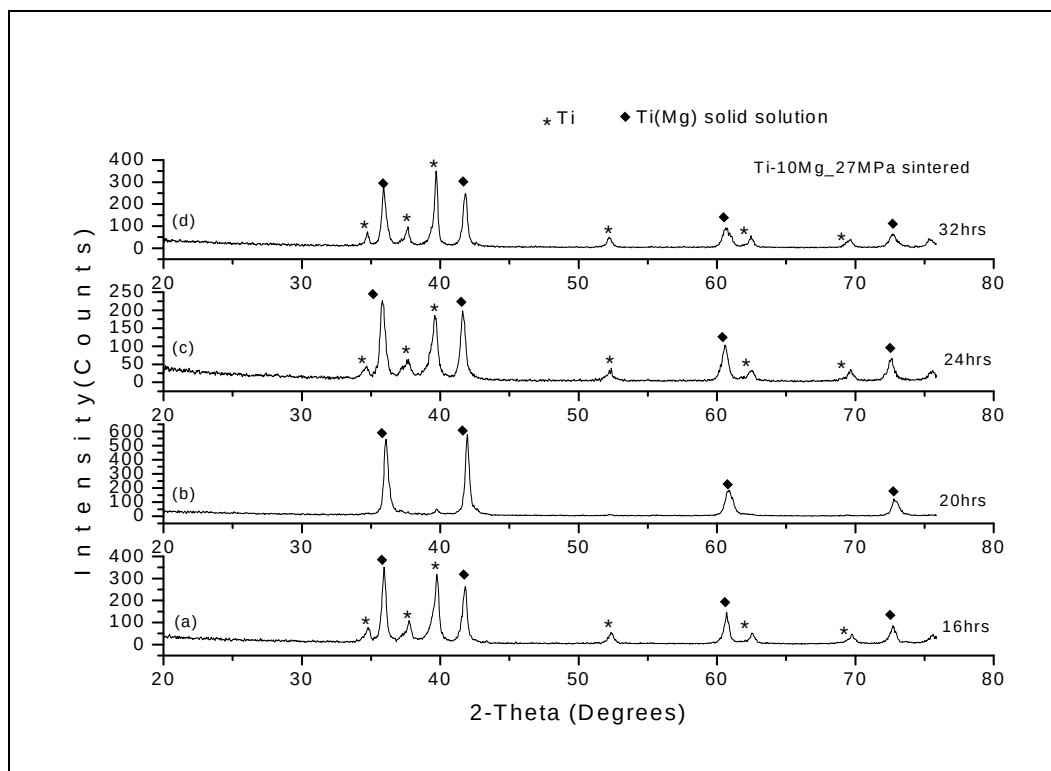


Figure 4.16: XRD spectra of sintered Ti-10Mg compacts pressed at 27 MPa

The evolution of phases in the sintered Ti-10Mg parts pressed at 33 MPa was similar to that of the parts that had been pressed at 27 MPa (**Figure 4.17**). However, the sample obtained from powder milled for 16 hours had a peak at

about 29.8° (2Theta) that could not be identified although it is suspected that it belongs to a Ti-oxide [51].

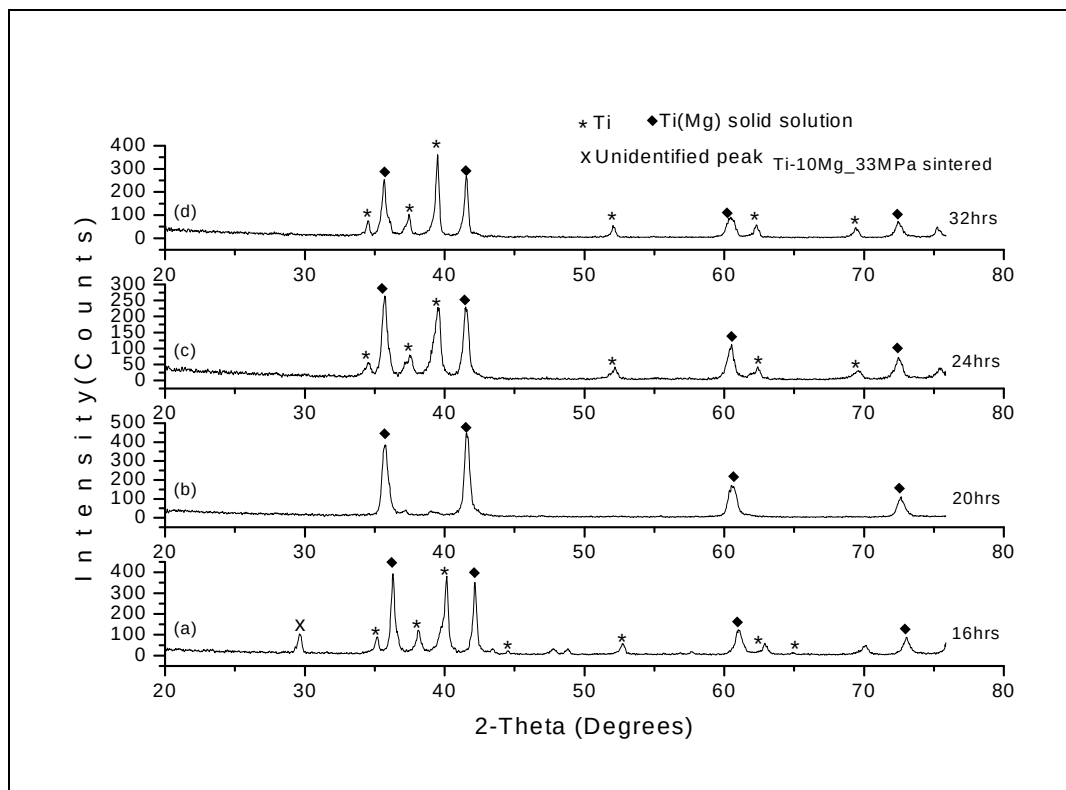


Figure 4.17: XRD spectra of sintered Ti-10Mg compacts pressed at 33 MPa.

4.3.1.2 XRD for Sintered Ti-20Mg compacts

XRD patterns for Ti-20Mg specimens pressed at both 27 MPa and 33 MPa were similar which implies that similar phases were formed (**Figures 4.18 and 4.19**). Peaks were sharper compared to those of the milled powders (**Figure 3.2**). This has been attributed to an increase in grain size due to coarsening

and also some Mg coming out of solution. Ti(Mg) solid solution peaks were identified in all the specimens. Ti peaks were also observed and arose from the decomposition of the Ti(Mg) phase.

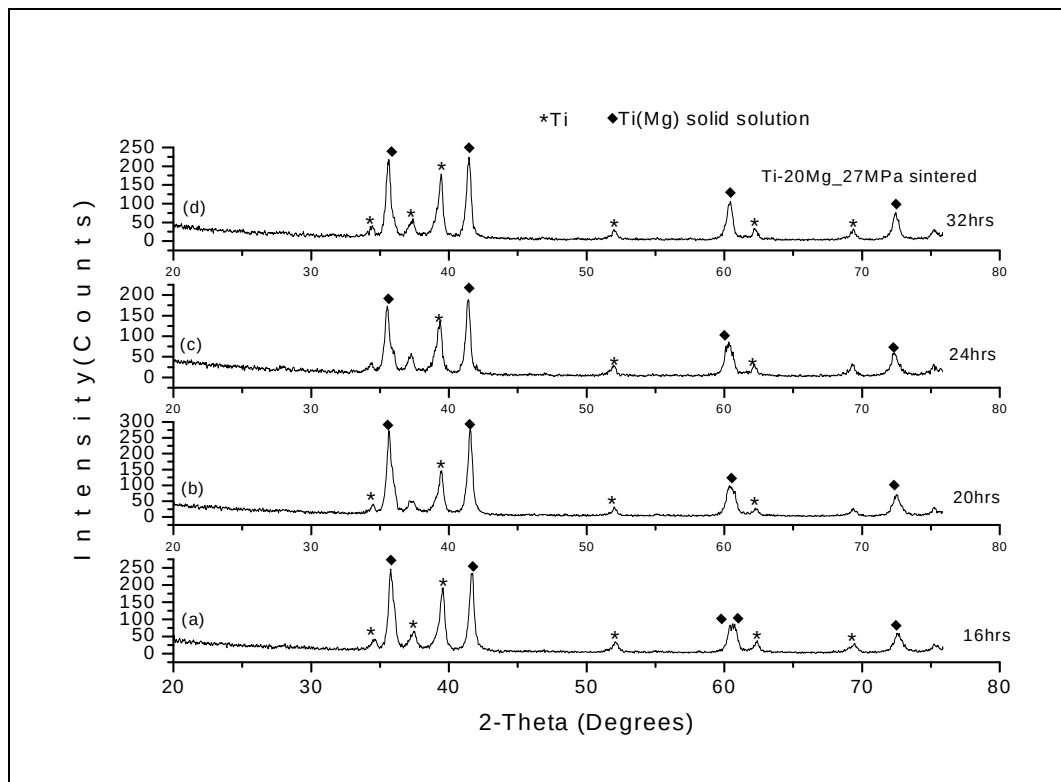


Figure 4.18: XRD spectra of sintered Ti-20Mg compacts pressed at 27 MPa

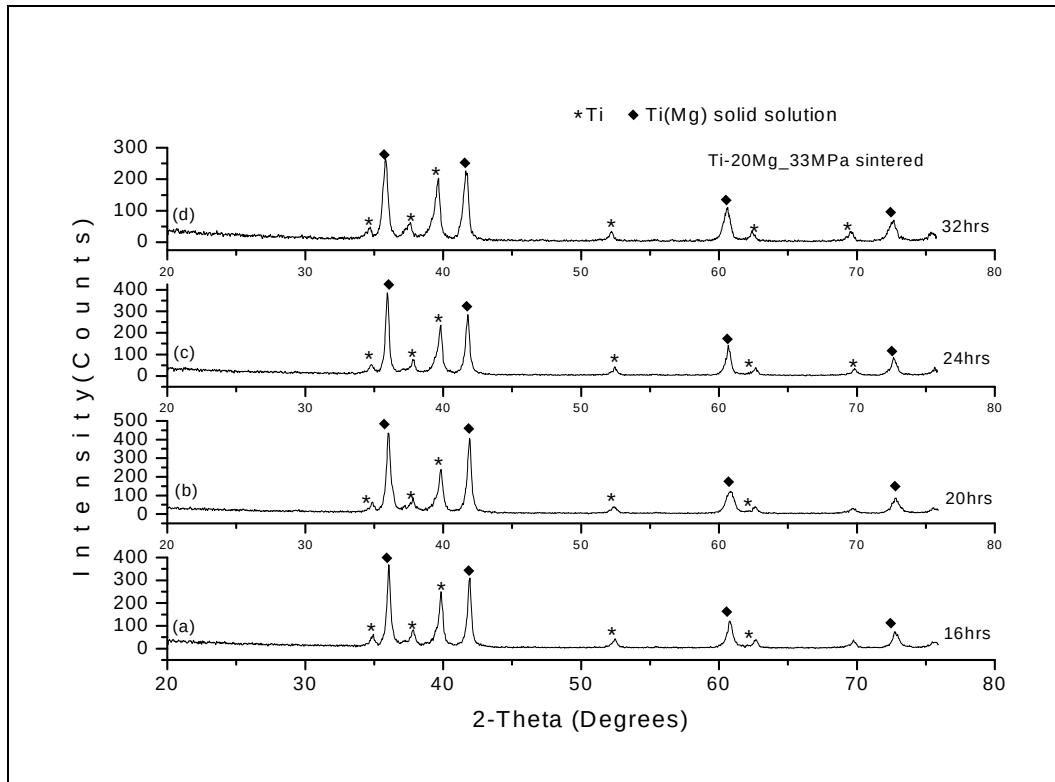


Figure 4.19: XRD spectra of sintered Ti-20Mg compacts pressed at 33 MPa

4.3.1.3 XRD for Sintered Ti-6Al-4Mg compacts

XRD patterns for the sintered Ti-6Al-4Mg samples are shown in **Figure 4.20** and **4.21** for the powders compacted with 27 and 33 MPa, respectively. The major peaks on the patterns are for Ti(Mg,Al) solid solution, which has been retained from the milled powders (Cf *Figure 4.5*). There are also peaks for Ti, indicating that the Ti(Mg,Al) solid solution underwent some decomposition. Compared to the milled powders, the peaks of the sintered samples are sharper, and shifted to the right with milling time. The pattern of the sample obtained from powder milled for 20 hours and pressed at 27 MPa (**Figure**

4.20b) has fewer peaks than the other patterns, indicating that maybe, the sintering was not successful.

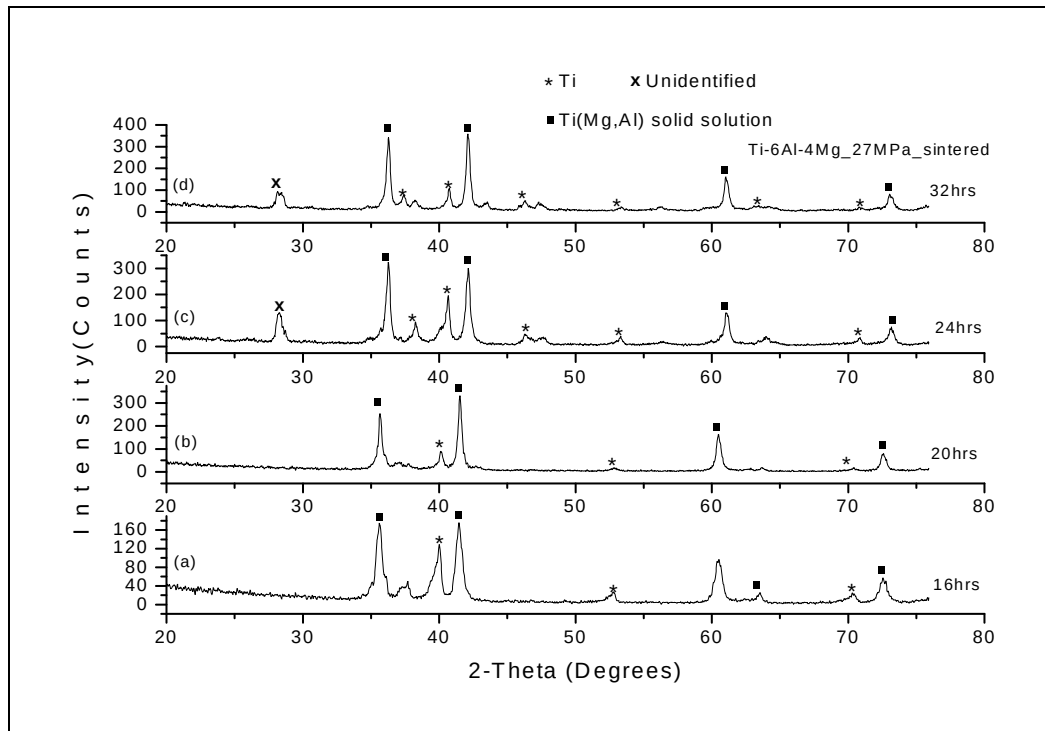


Figure 4.20: XRD spectra of sintered Ti-6Al-4Mg compacts pressed at 27MPa

A new peak at $2\theta=28^\circ$ was observed in sintered compacts from powders milled for 24 and 32 hours and pressed with 27 MPa and for all the powders pressed with 33 MPa. This peak could not be matched but it is suspected that it is for a Ti-oxide.

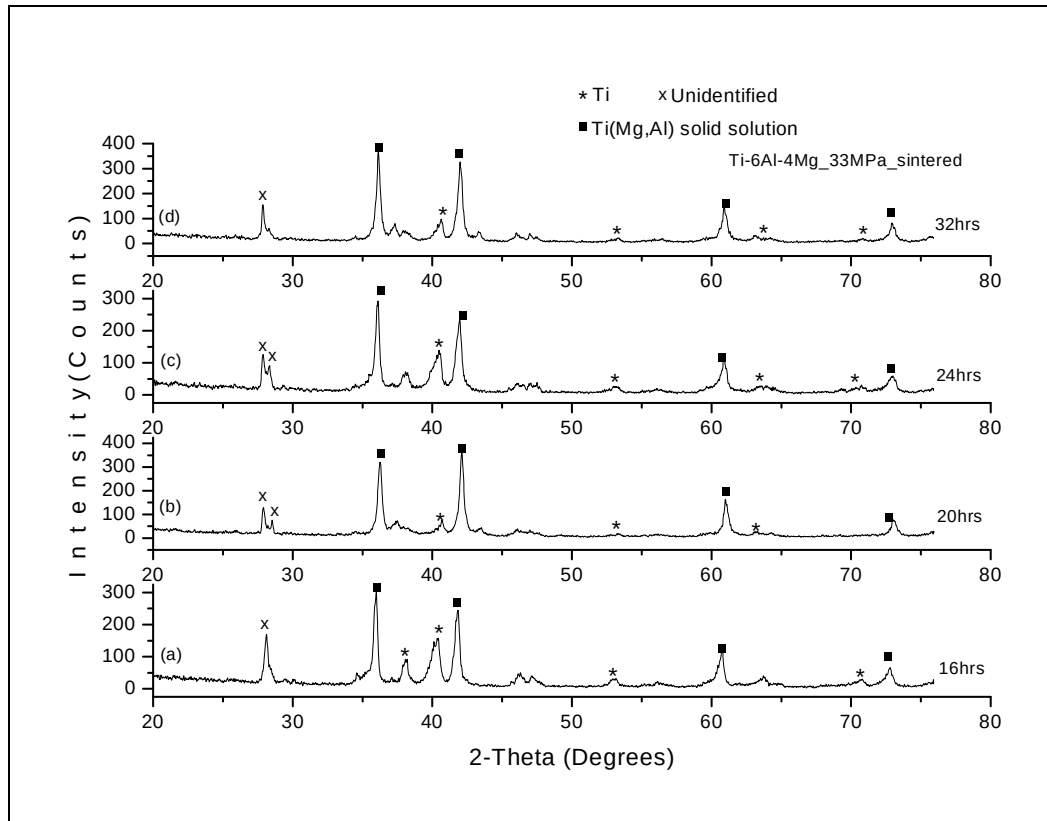


Figure 4.21: XRD spectra of sintered Ti-6Al-4Mg compacts pressed at 33 MPa.

4.3.1.4 Summary of phases in sintered parts

Tables 9 and **10** summarize the phases present in the sintered Ti-10Mg and Ti-6Al-4Mg specimens. All Ti-20Mg specimens revealed the presence of similar phases namely Ti(Mg) and Ti. The results for Ti-6Al-4Mg also suggest that longer milling times and higher compaction pressures induce the formation of the oxide phase.

Table 9: Phases present in sintered Ti-10Mg specimens

Specimen	Phases present
Ti-10Mg_16h_27MPa	Ti(Mg), Ti
Ti-10Mg_20h_27MPa	Ti(Mg)
Ti-10Mg_24h_27MPa	Ti(Mg), Ti
Ti-10Mg_32h_27MPa	Ti(Mg), Ti
Ti-10Mg_16h_33MPa	Ti(Mg), Ti, X
Ti-10Mg_20h_33MPa	Ti(Mg)
Ti-10Mg_24h_33MPa	Ti(Mg), Ti
Ti-10Mg_32h_33MPa	Ti(Mg), Ti

Table 10: Phases present in sintered Ti-6Al-4Mg specimens

Specimen	Phases present
Ti-6Al-4Mg_16h_27MPa	Ti(Mg,Al), Ti,
Ti-6Al-4Mg_20h_27MPa	Ti(Mg,Al), Ti
Ti-6Al-4Mg_24h_27MPa	Ti(Mg,Al), Ti, X
Ti-6Al-4Mg_32h_27MPa	Ti(Mg,Al), Ti, X
Ti-6Al-4Mg_16h_33MPa	Ti(Mg,Al), Ti, X
Ti-6Al-4Mg_20h_33MPa	Ti(Mg,Al), Ti X
Ti-6Al-4Mg_24h_33MPa	Ti(Mg,Al), Ti, X
Ti-6Al-4Mg_32h_33MPa	Ti(Mg,Al), Ti, X

X- Unidentified phase suspected to be a Ti_xO_y phase

4.3.2 Microstructure (Optical and SEM Micrographs)

Most specimens were soft which made surface preparation difficult, hence the presence of polishing lines. Optical micrographs of the sintered specimens generally showed the occurrence of dark/black spots that are usually associated with porosity [33] and unsintered powder. The most porous samples were those obtained from Ti-10Mg and Ti-6Al-4Mg powders milled for 20 hours. These powders might not have pressed well because they had a higher content of fines (**Figures 4.1 and 4.8**). The darker region was observed to be proportionally larger in samples which did not sinter well. Back-scattered and secondary electron images (SEI) have also been added to complement optical micrographs.

4.3.2.1 Ti-10Mg specimens

Figure 4.22 – 4.23 show optical micrographs of Ti-10Mg specimens pressed at 27 MPa. The major observable difference was on the samples from compacts from powders milled for 20 hours (**Figure 4.22 b**) which were differentiated by a continuous dark matrix. The sectioned surface of this specimen did not reveal a metallic finish which shows that it did not sinter well and the continuous dark matrix represent extensive and interconnected porosity. If this is so, relatively low hardness values can be expected in alloys with more of the “darker phase”. The other specimens were relatively less porous and they are expected to possess relatively higher hardness values. In fact, better physical properties such as density can be expected.

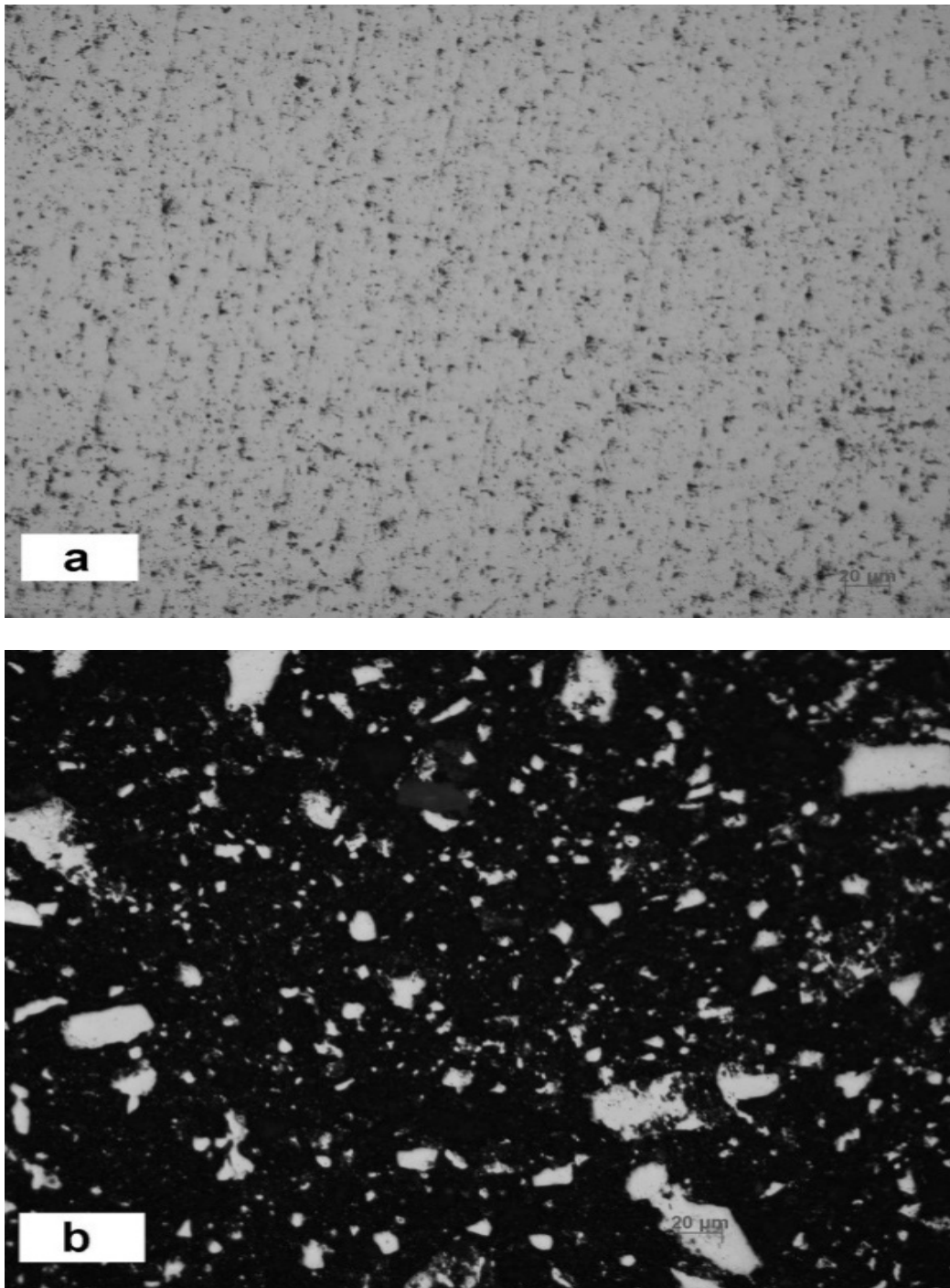


Figure 4.22: Micrographs for Ti-10Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h

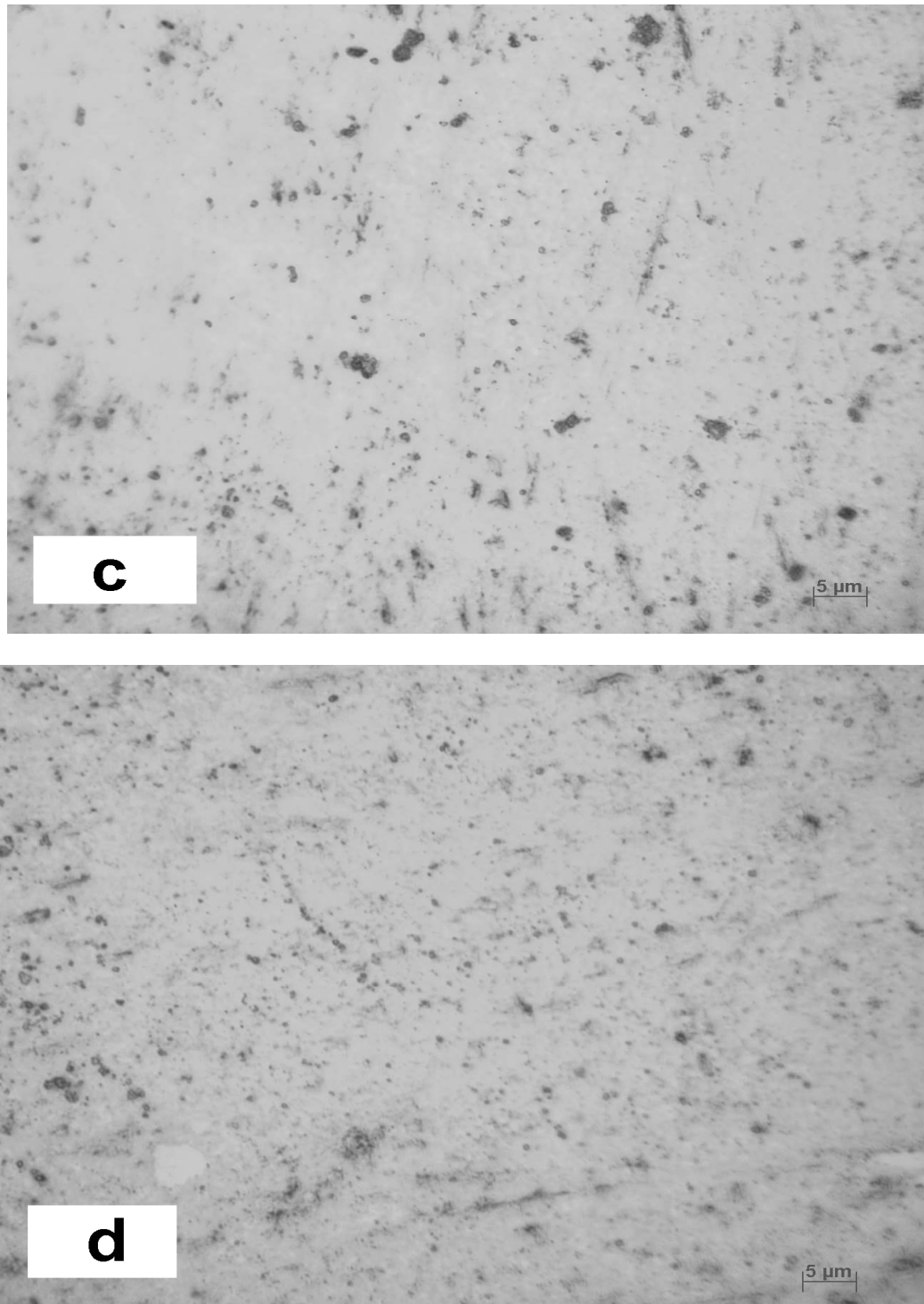


Figure 4.23: Micrographs for Ti-10Mg specimens pressed at 27 MPa from powders milled for (c) 24 h (d) 32 h

Figure 4.24 shows a characteristic high magnification optical micrograph of the specimens.

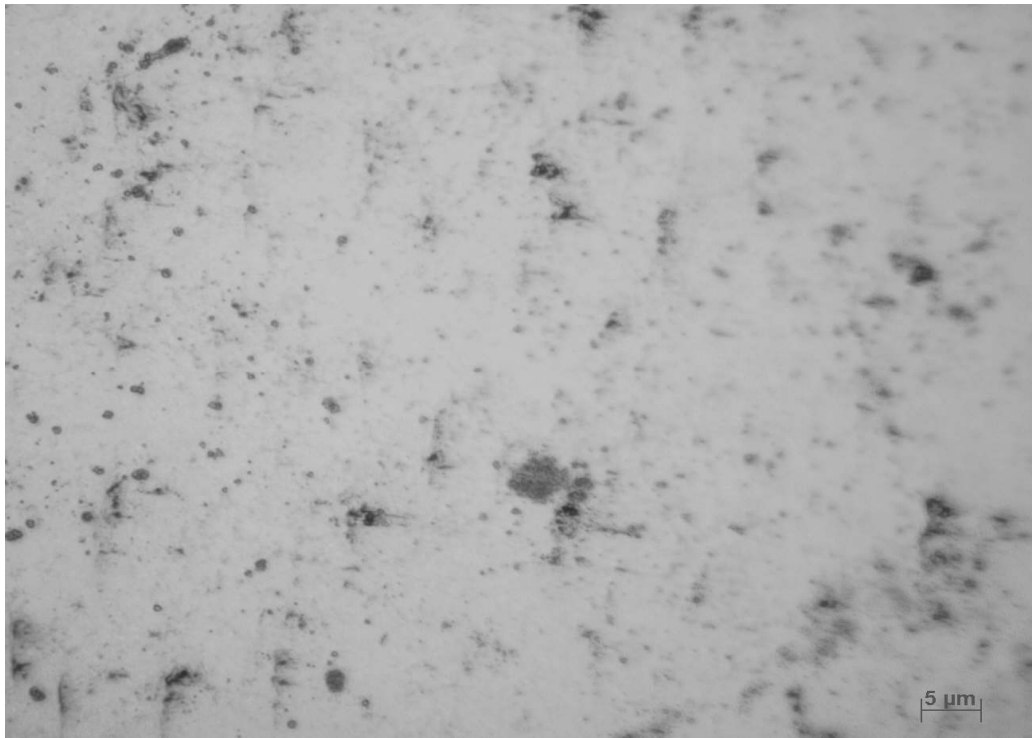


Figure 4.24: Micrograph of a Ti-10Mg specimen from powders milled for 16 hours and pressed at 27 MPa

Optical micrographs for specimens pressed at 33 MPa are shown in **Figures 4.25 – 4.26**. The Ti-10Mg_20h_33 MPa specimen (**Figure 4.26b**) shows a similar microstructure to **Figure 4.22b**. Porosity in these specimens is attributed to the higher content of fines in the milled powders; thus the powders might not have pressed well. Green density results for these specimens were also relatively low (**See Figure 4.11**). The porosity of the other samples was relatively low.

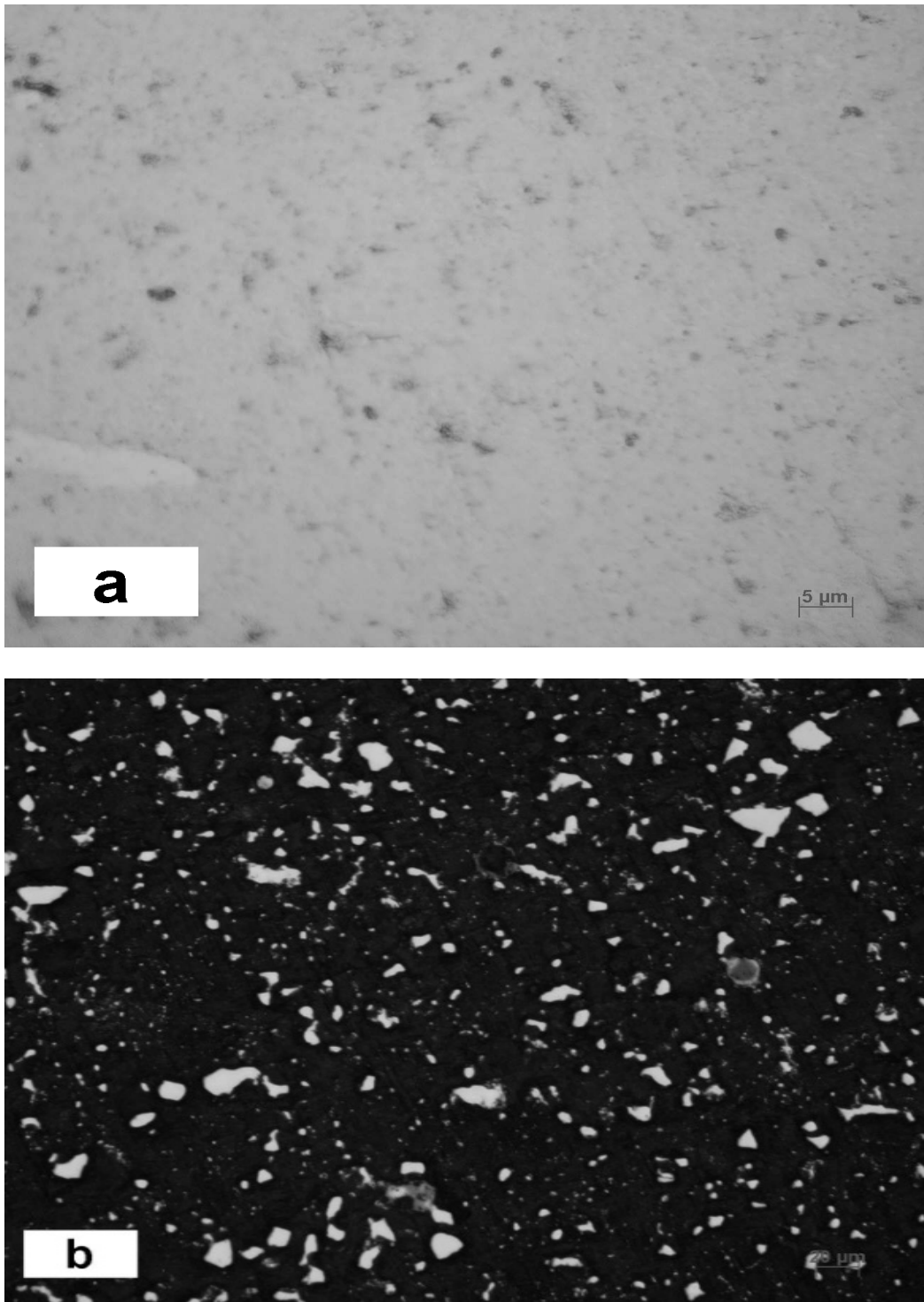


Figure 4.25: Micrographs for Ti-10Mg specimens pressed at 33 MPa from powders milled for (a) 16 h (b) 20 h

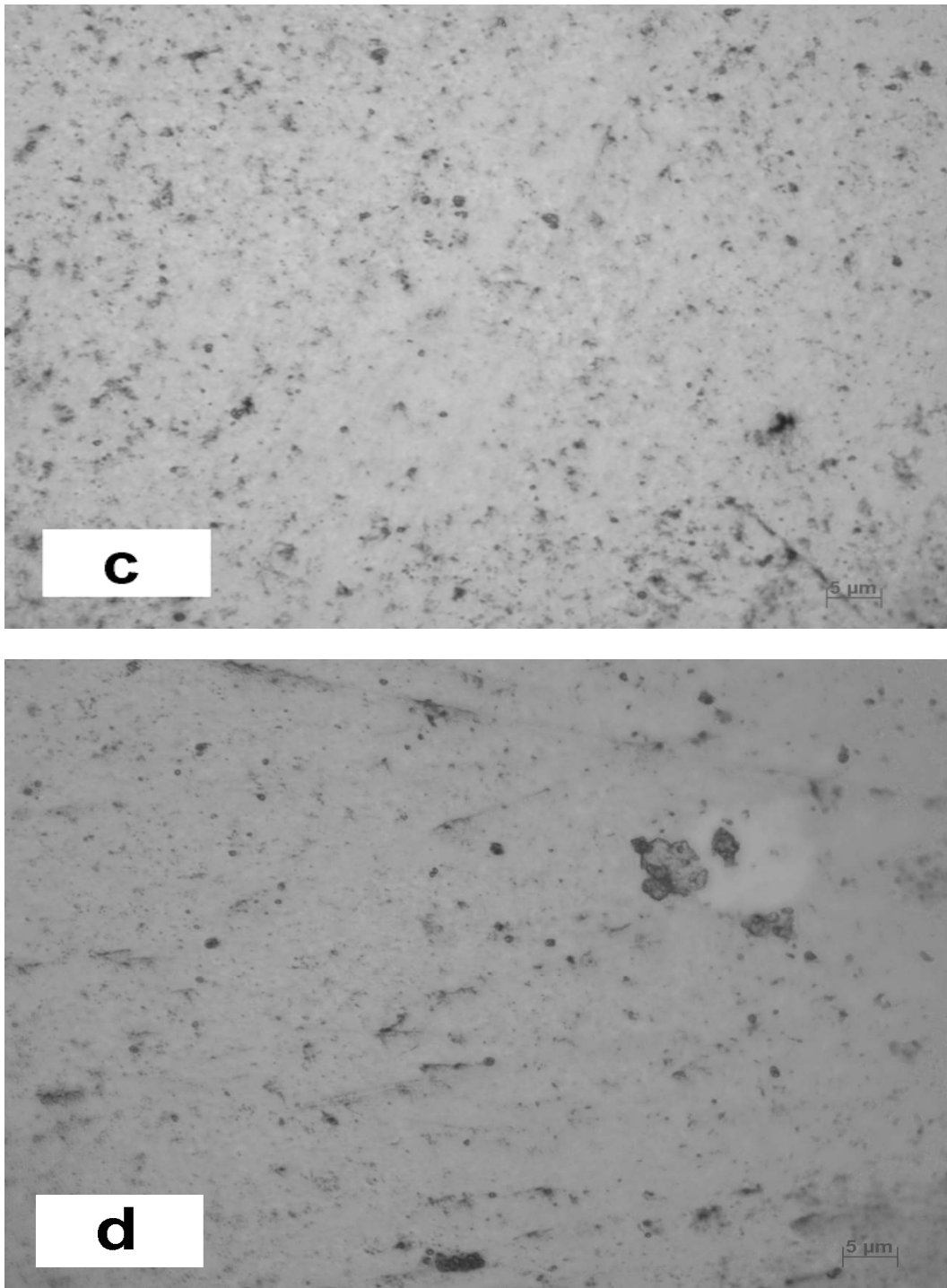


Figure 4.26: Micrographs for Ti-10Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h.

The SEM images for Ti-10Mg specimens from powders compacted at 27 and 33 MPa are shown in **Figures 4.27 - 4.30**. The images also reveal porosity. Ti-10Mg compacts from powders milled for 20 hours exhibited extensive and interconnected porosity (**Figures 4.27 b** and **4.29 b**). Similar results were observed in the optical micrographs (**Figure 4.25 b**). The images also show reduced porosity at higher compaction pressure. Grey and lighter regions were observed in the images. EDS analysis revealed that the darker or grayish regions were Mg-rich areas while the lighter regions were Ti-rich.

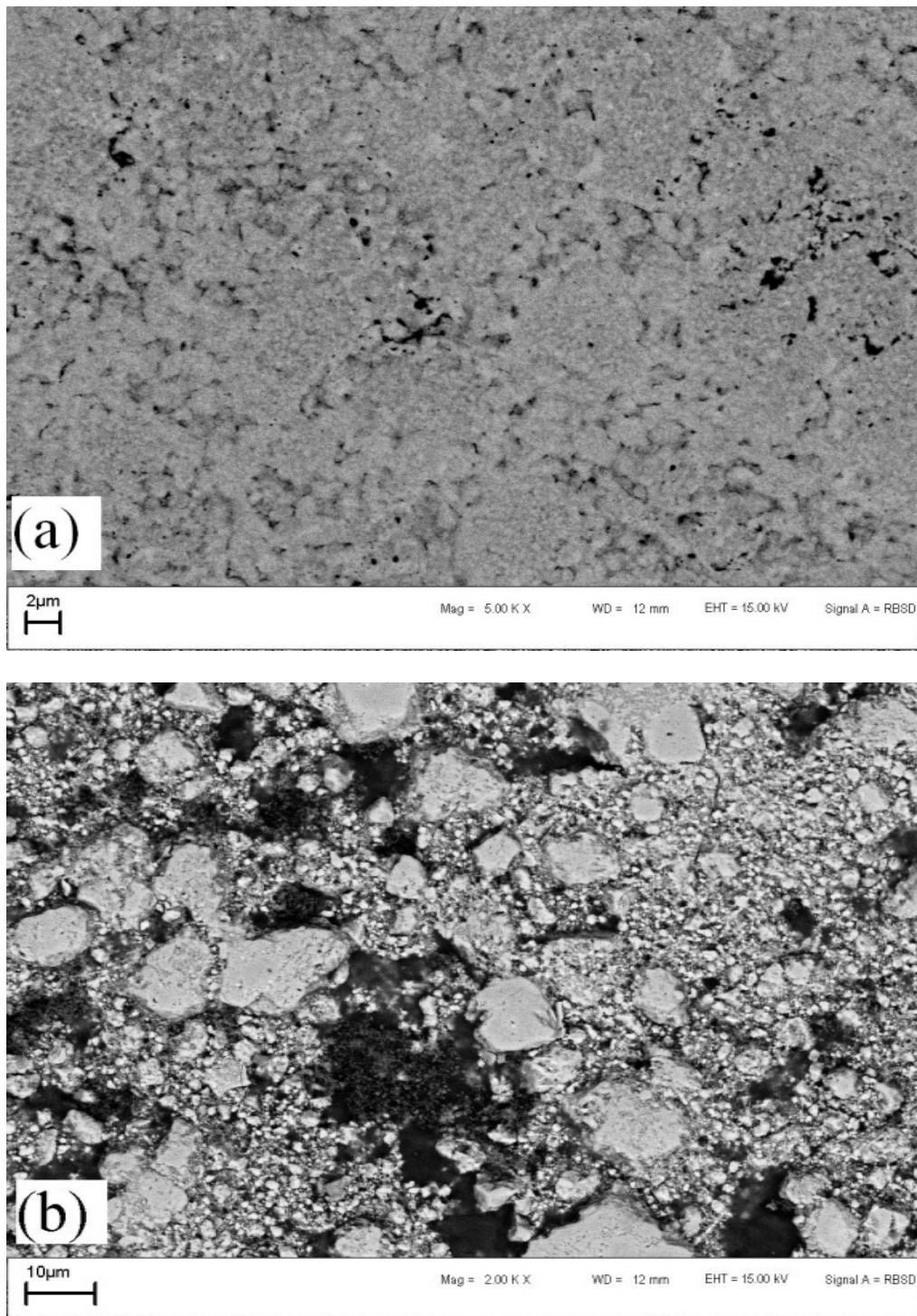


Fig 4.27: SEM Images of Ti-10Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h

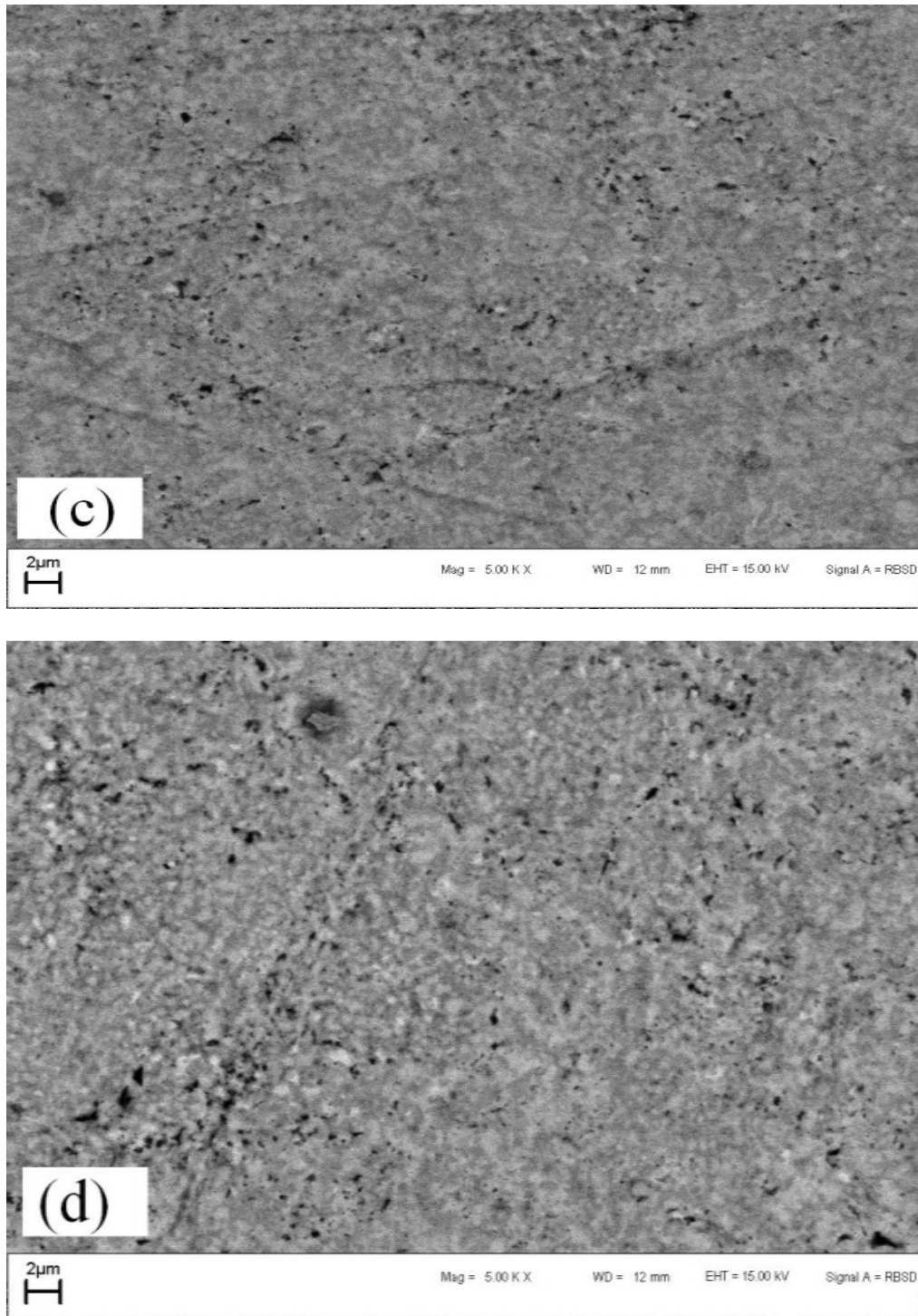


Fig 4.28: SEM Images of Ti-10Mg specimens pressed at 27 MPa from powders milled for (c) 24 h (d) 32 h.

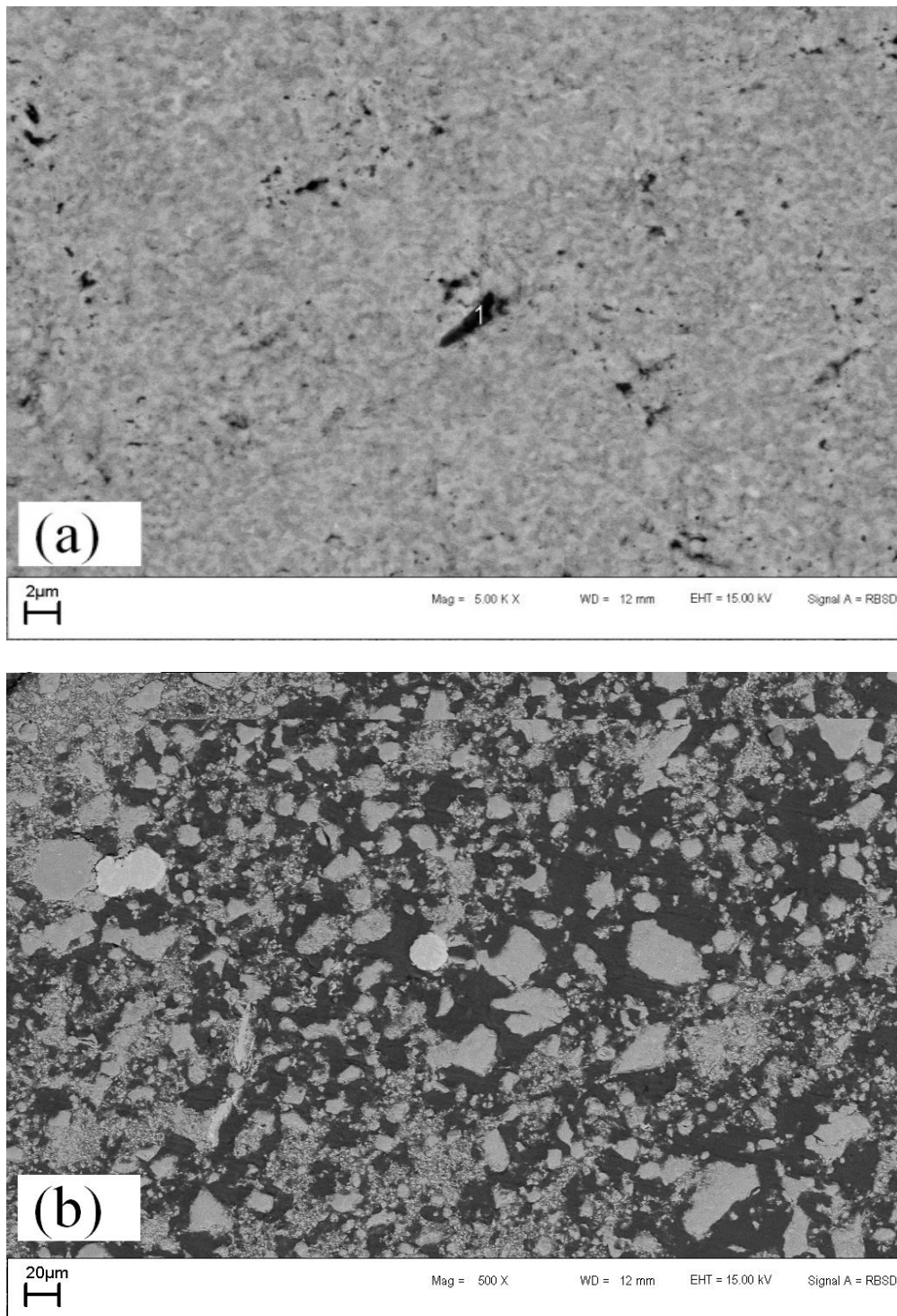


Fig 4.29: SEM Images of Ti-10Mg specimens pressed at 33 MPa from powders milled for (a) 16 h (b) 20 h

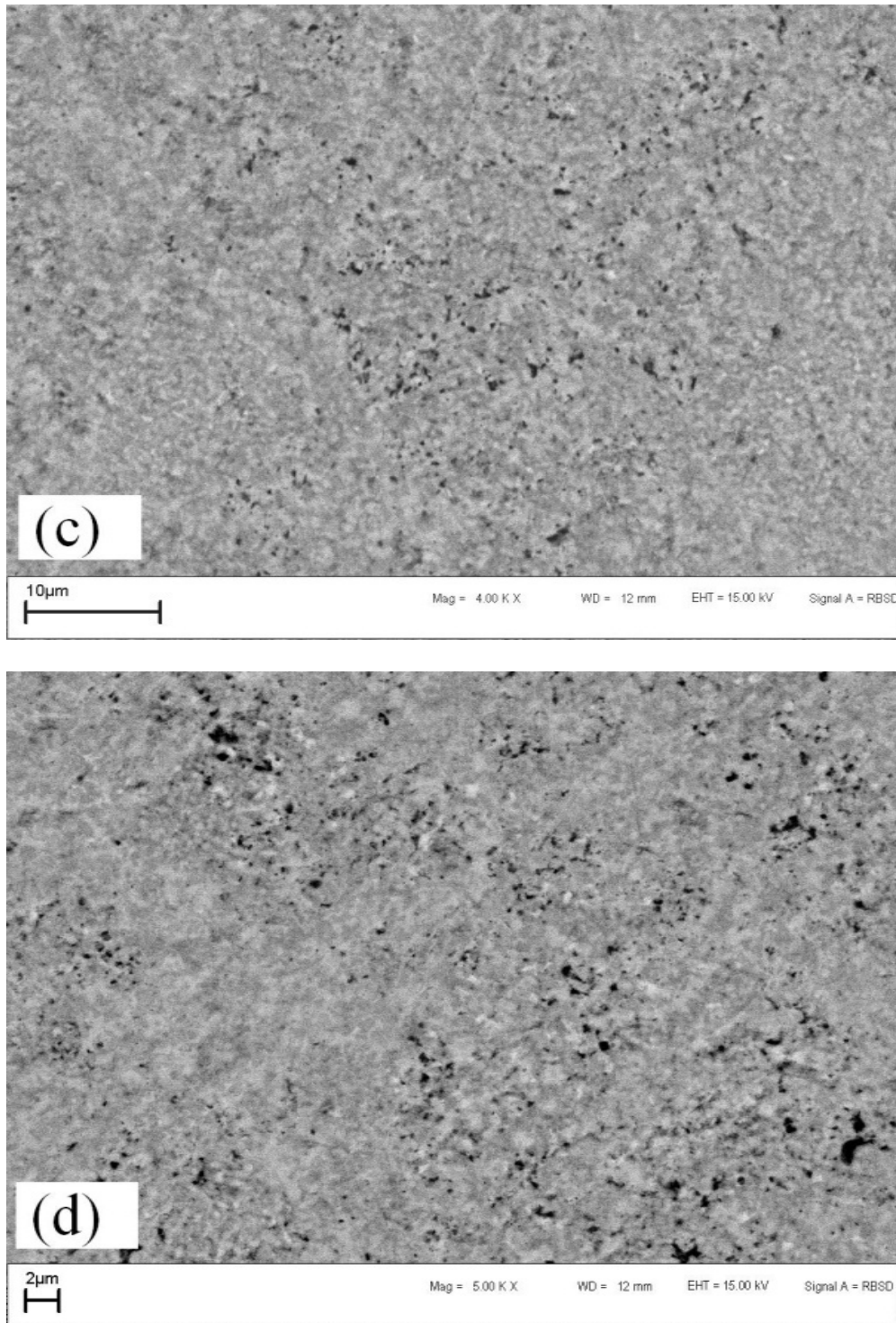


Fig 4.30: SEM Images of Ti-10Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h.

4.3.2.2 Ti-20Mg specimens

Ti-20Mg specimens revealed microstructures in which the level of porosity was dependent on the compaction pressure (**Figures 4.31 - 4.34**). Compacts pressed at 33 MPa are less porous than those pressed at 27 MPa. This is expected since these compacts had relatively higher green densities.

Figures 4.36 - 4.39 show the corresponding SEM images for Ti-20Mg samples pressed at 27 and 33 MPa. The specimens were porous but the porosity is less marked compared to those of Ti-10Mg that were obtained from powders milled for 20 hours (e.g. **Figure 4.22 (b)**). This suggests that the powder particle size distribution of the Ti-20Mg powders was amenable to cold compaction. Porosity in the Ti-20Mg samples appeared to decrease with increasing milling time and increasing compaction pressure. There is also an indication of the Mg quantity having an influence on porosity.

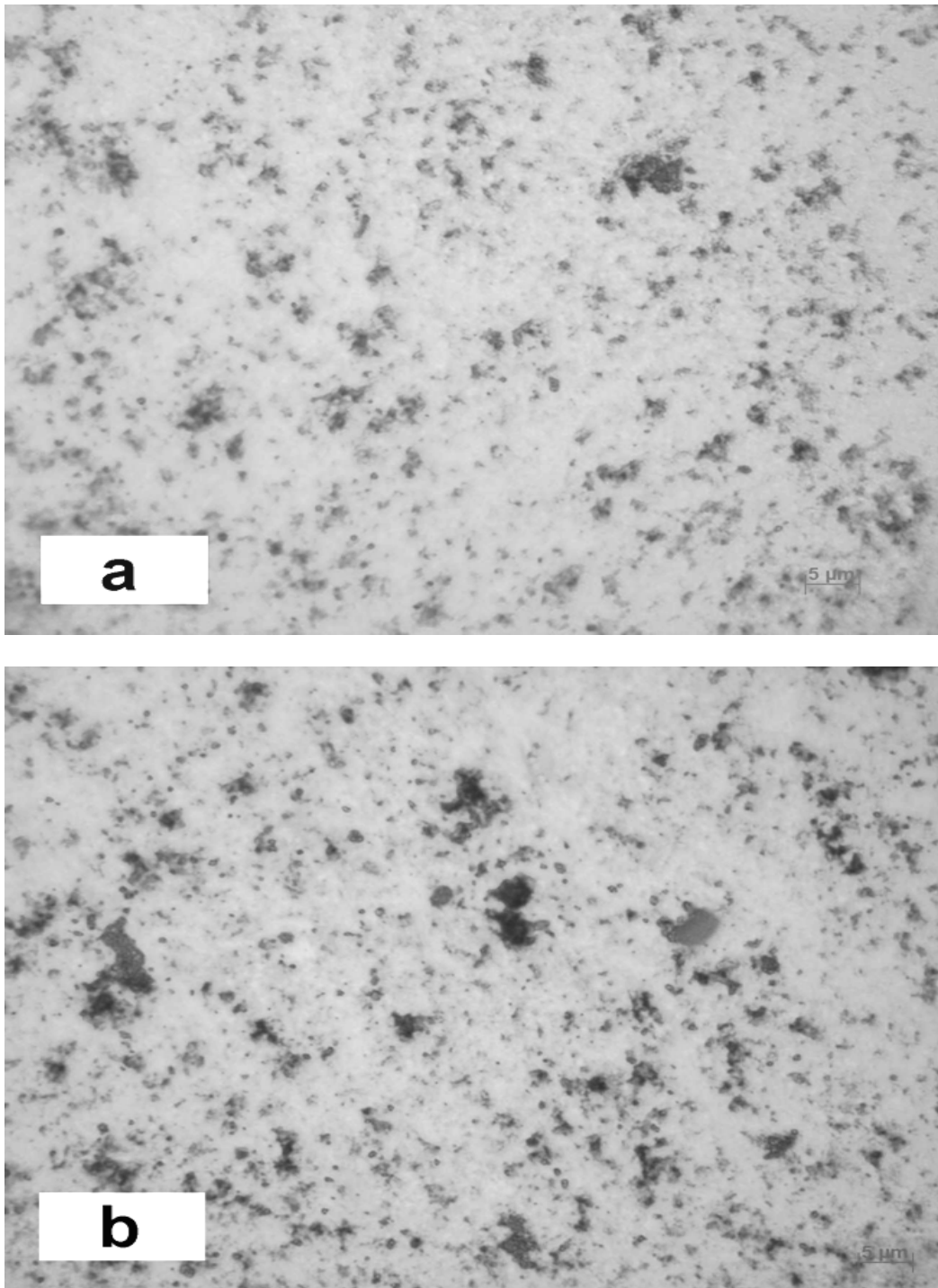


Figure 4.31: Micrographs for Ti-20Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h.

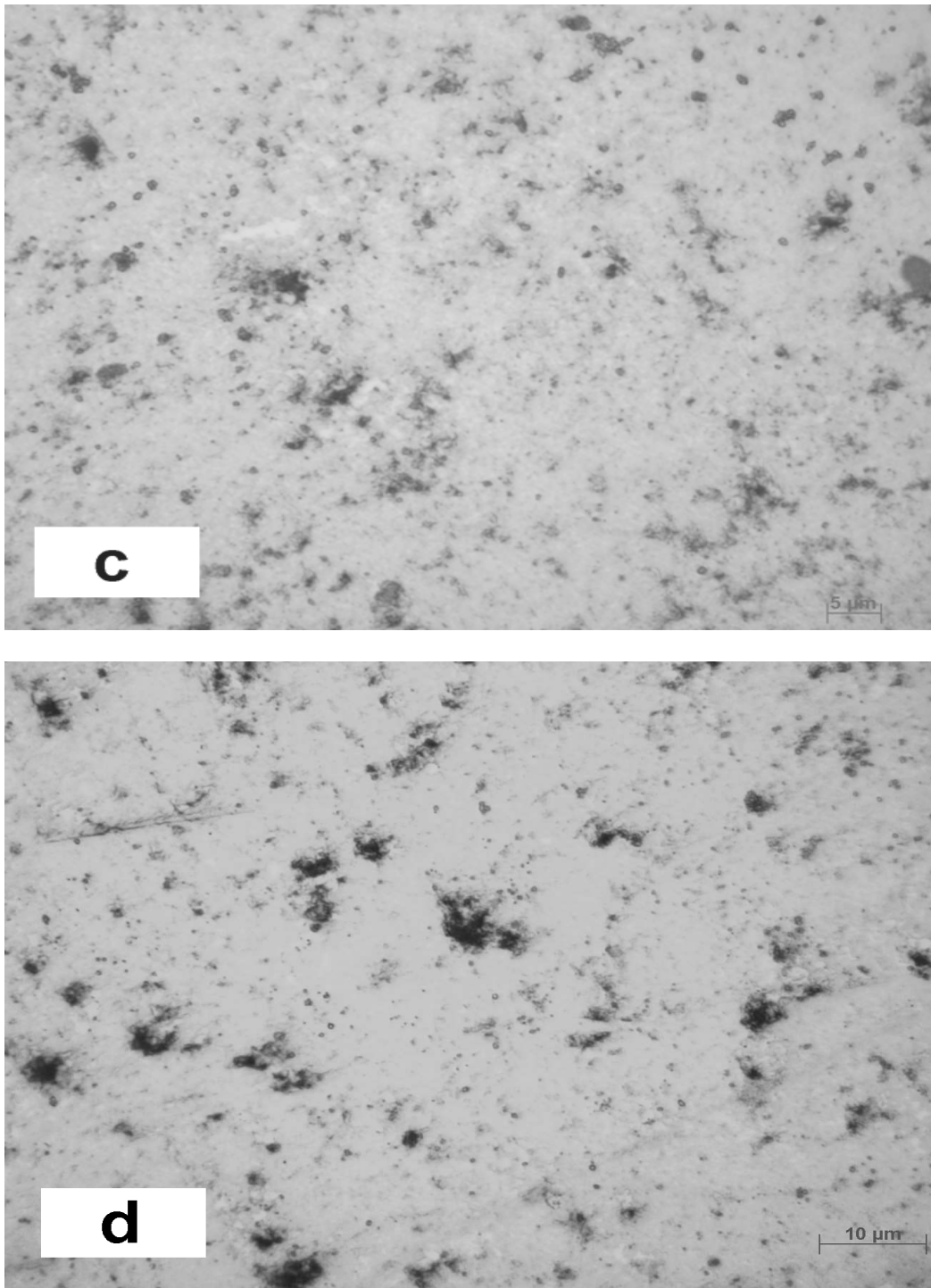


Figure 4.32: Micrographs for Ti-20Mg specimens pressed at 27 MPa from powders milled for (c) 24 h (d) 32 h.

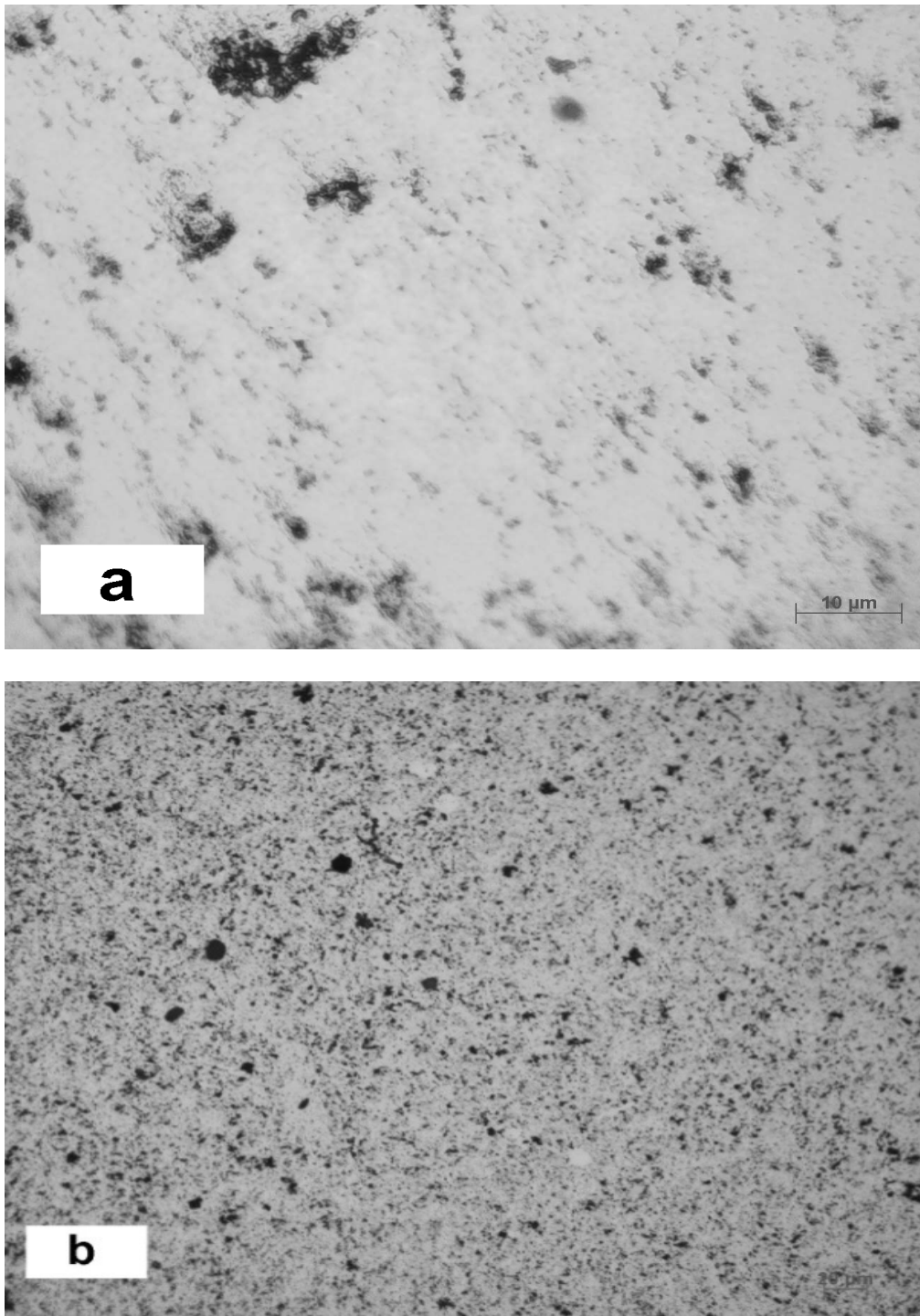


Figure 4.33: Micrographs for Ti-20Mg specimens pressed at 33 MPa from powders milled for (a) 16 h (b) 20 h

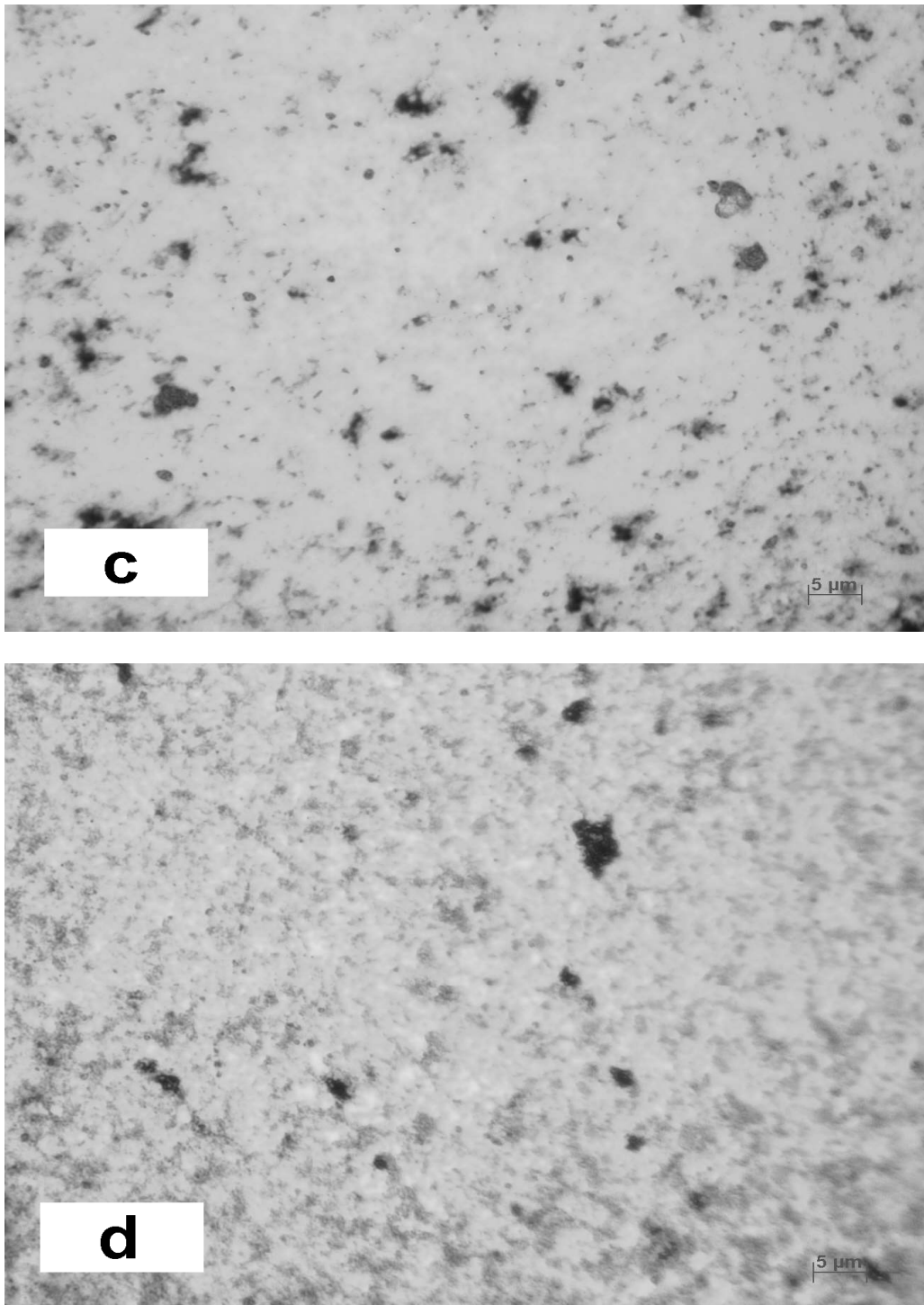


Figure 4.34: Micrographs for Ti-20Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h.

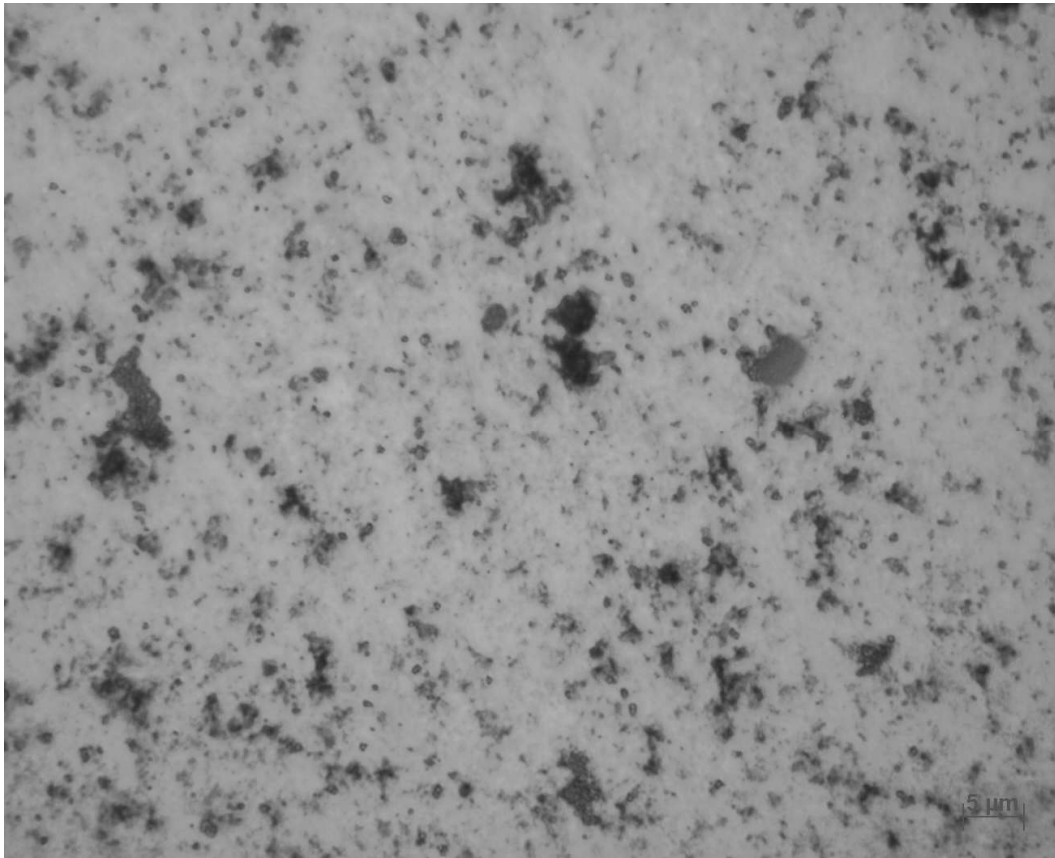


Figure 4.35: Micrograph of a Ti-20Mg specimen from powders milled for 16 hours and pressed at 33 MPa

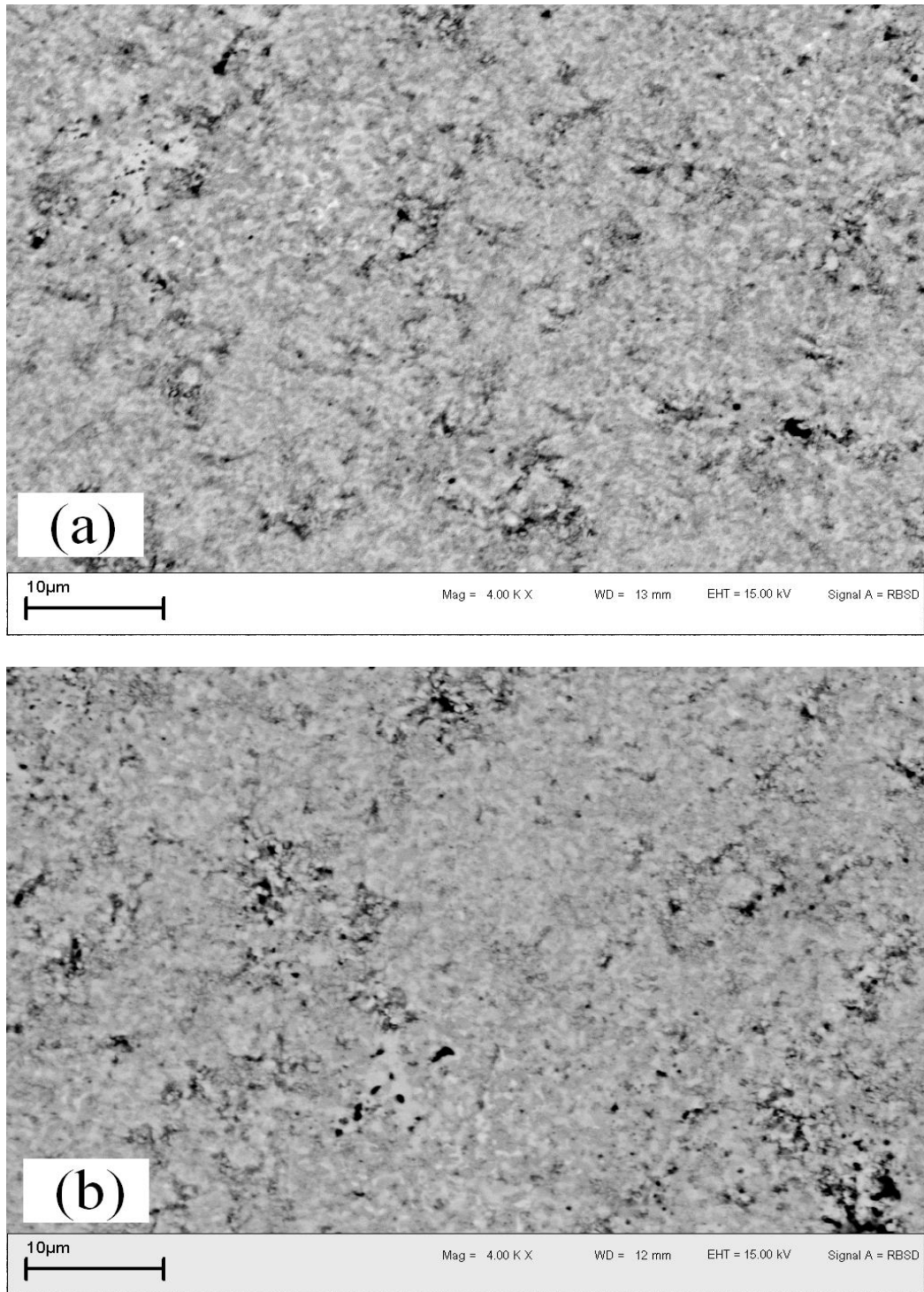


Figure 4.36: SEM Images of Ti-20Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h

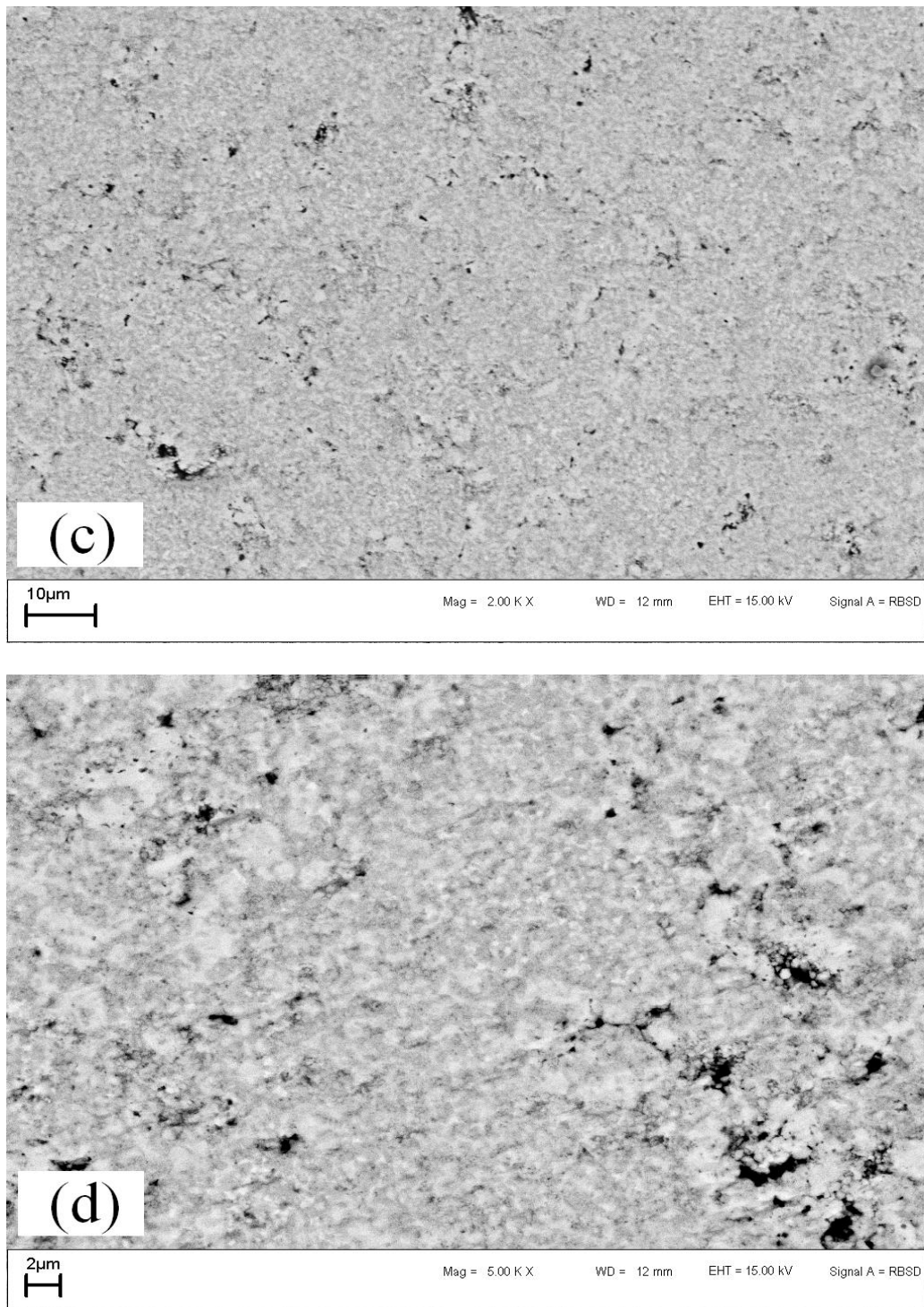


Figure 4.37: SEM Images of Ti-20Mg specimens pressed at 27 MPa from powders milled for (c) 24 h (d) 32 h.

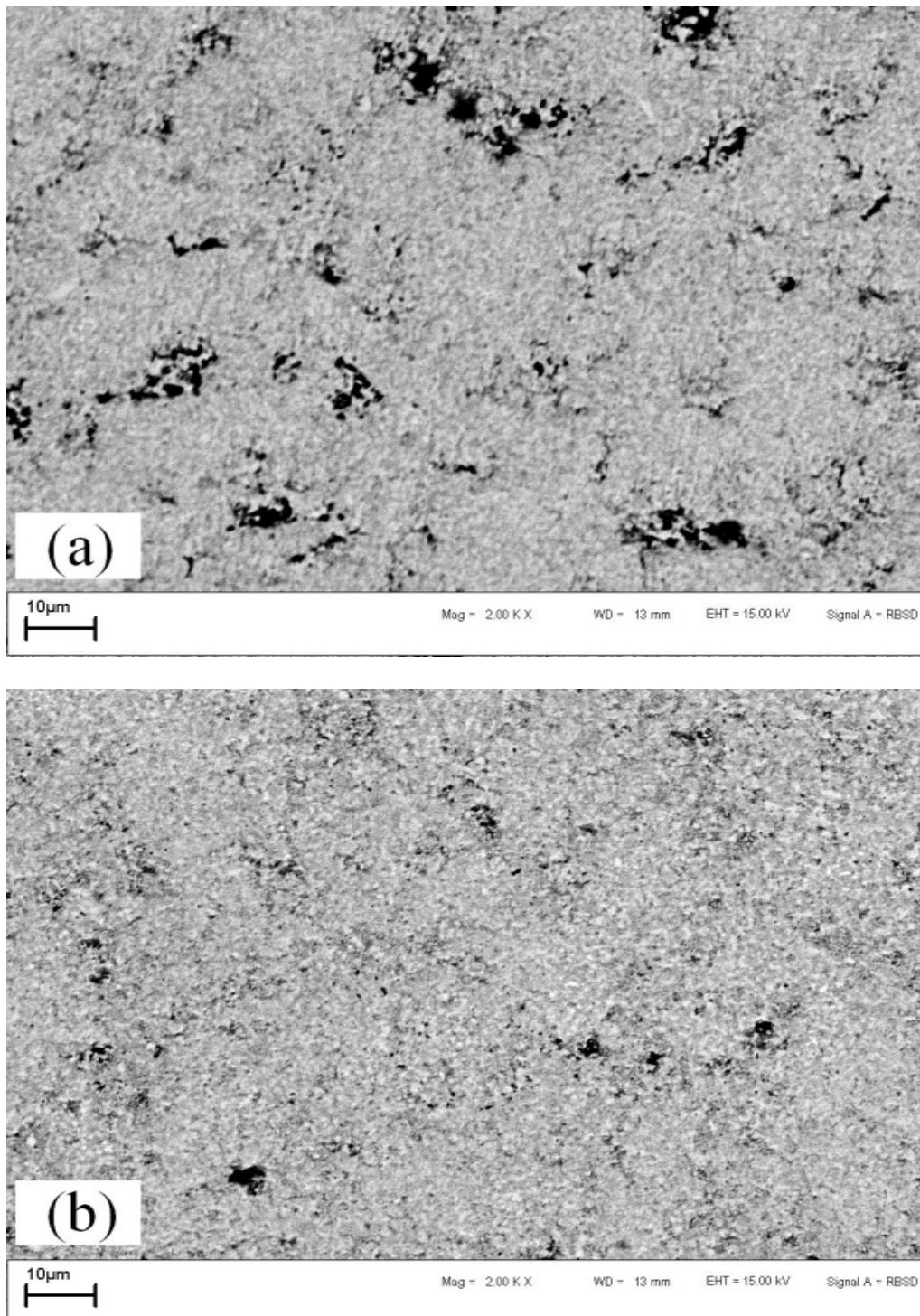


Figure 4.38: SEM Images of Ti-20Mg specimens pressed at 33 MPa from powders milled for (a) 16 h (b) 20 h.

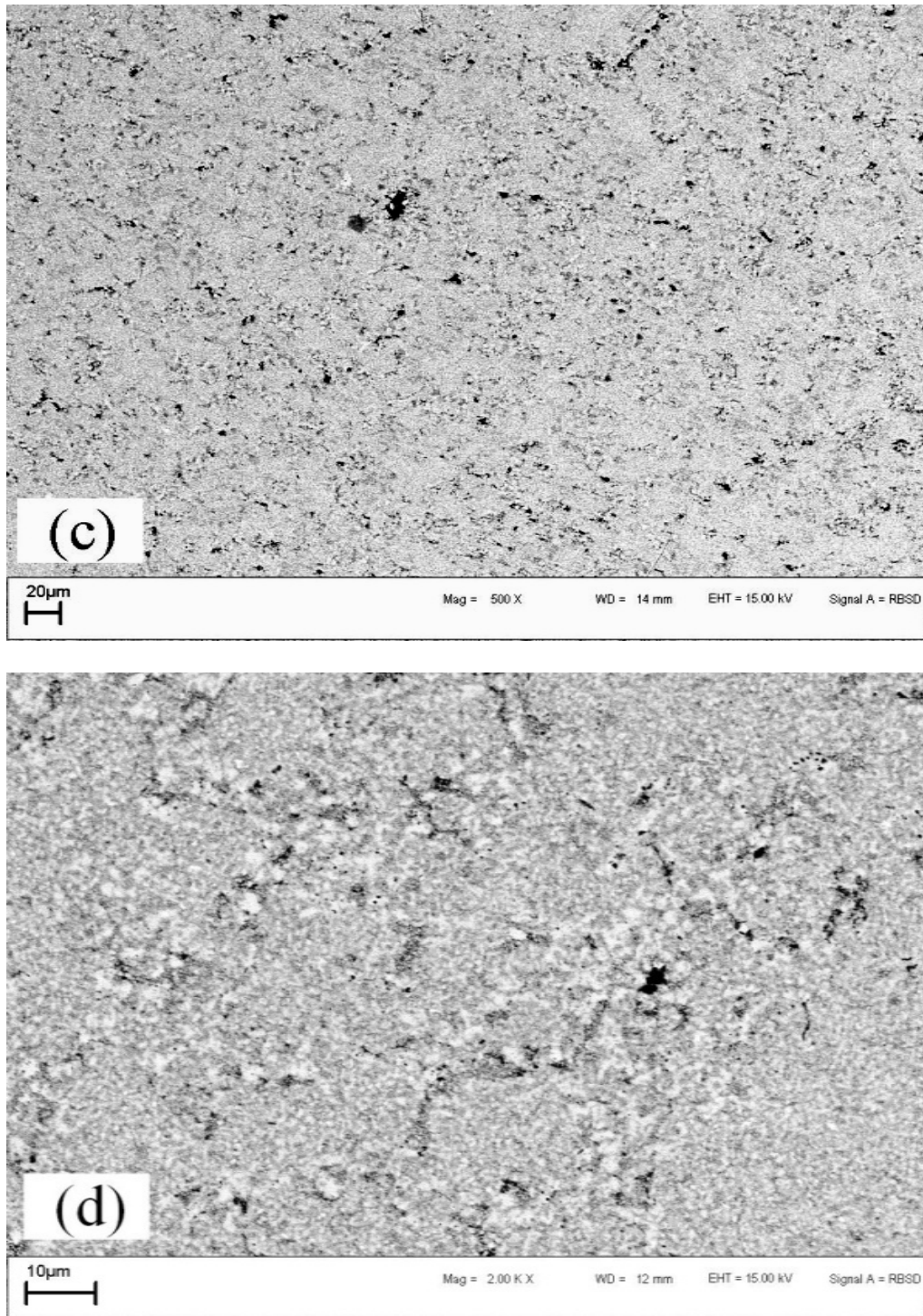


Figure 4.39: SEM Images of Ti-20Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h.

4.3.2.3 Ti-6Al-4Mg specimens

Figures 4.40 to 4.41 show optical micrographs for Ti-6Al-4Mg samples from powders pressed at 27 MPa. The microstructures were porous and the level of the porosity depended on the milling time. Specimen from powders milled for 20 hours (**Figures 4.40 b**) show extensive and interconnected porosity. This is attributed to the presence of a significant amount of fines in the milled powders and relatively low compaction pressure used. It is also possible that the presence of unsintered powder particles might be contributing to the high porosity observed.

Secondary electron (SE) images of Ti-6Al-4Mg specimens confirmed the observations of porosity in the specimens. The specimen obtained from powders milled for 20 hours and pressed at 27 MPa (**Figure 4.42 b**) was found to be very porous compared to the specimen pressed at 27 MPa from powders milled for 16, 24 and 32 hours (**Figure 4.42a and 4.43**). Since the XRD patterns show similar phases present, variation in porosity can be expected to affect properties such as hardness. Thus, very porous specimens are expected to record low hardness values.

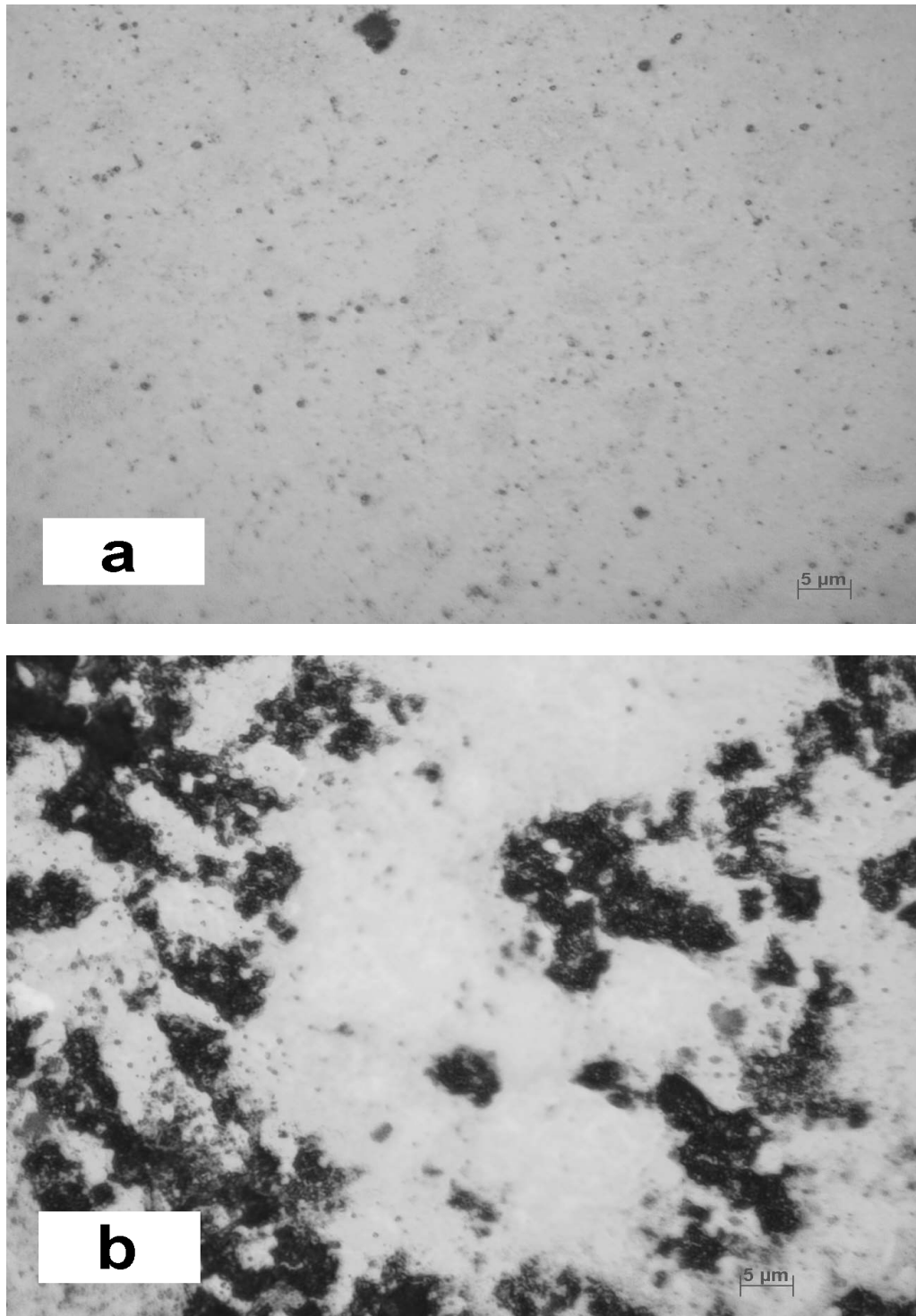


Figure 4.40: Micrographs for Ti-6Al-4Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h.

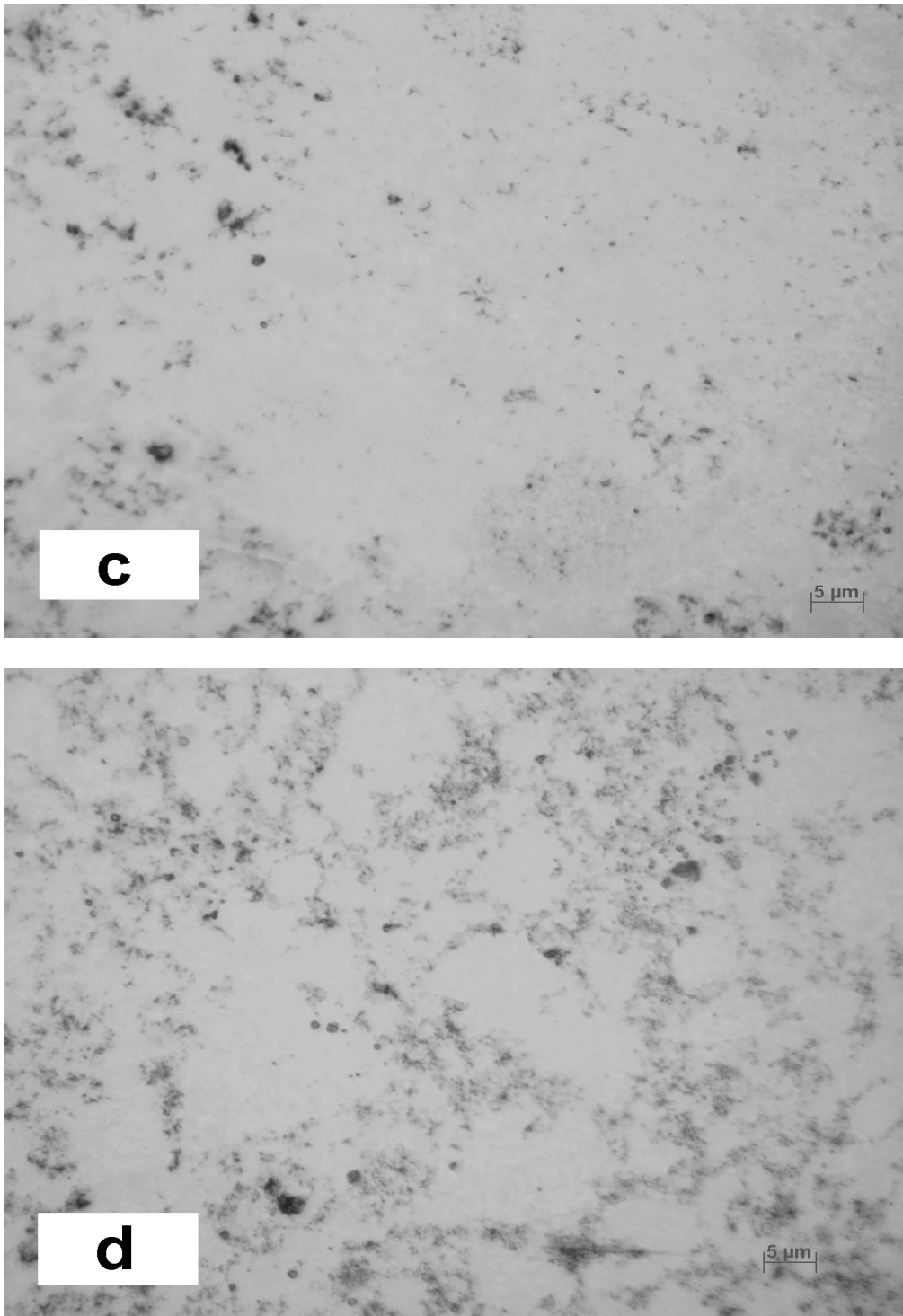


Figure 4.41: Micrographs for Ti-6Al-4Mg specimens pressed at 27 MPa from powders milled for (c) 24 h (d) 32 h.

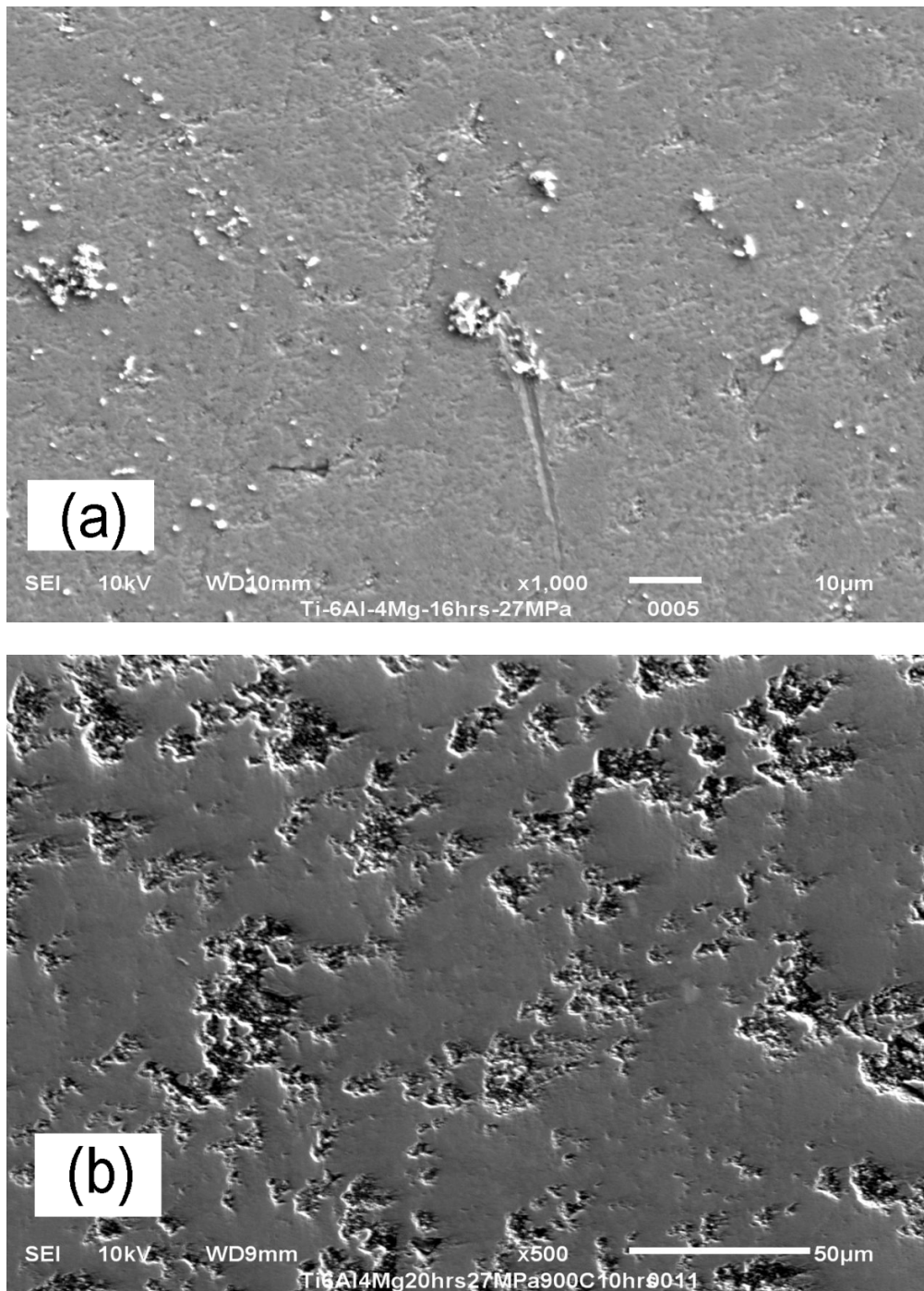


Figure 4.42: Secondary electron (SE) image of Ti-6Al-4Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h; with **b** showing extensive and interconnected porosity

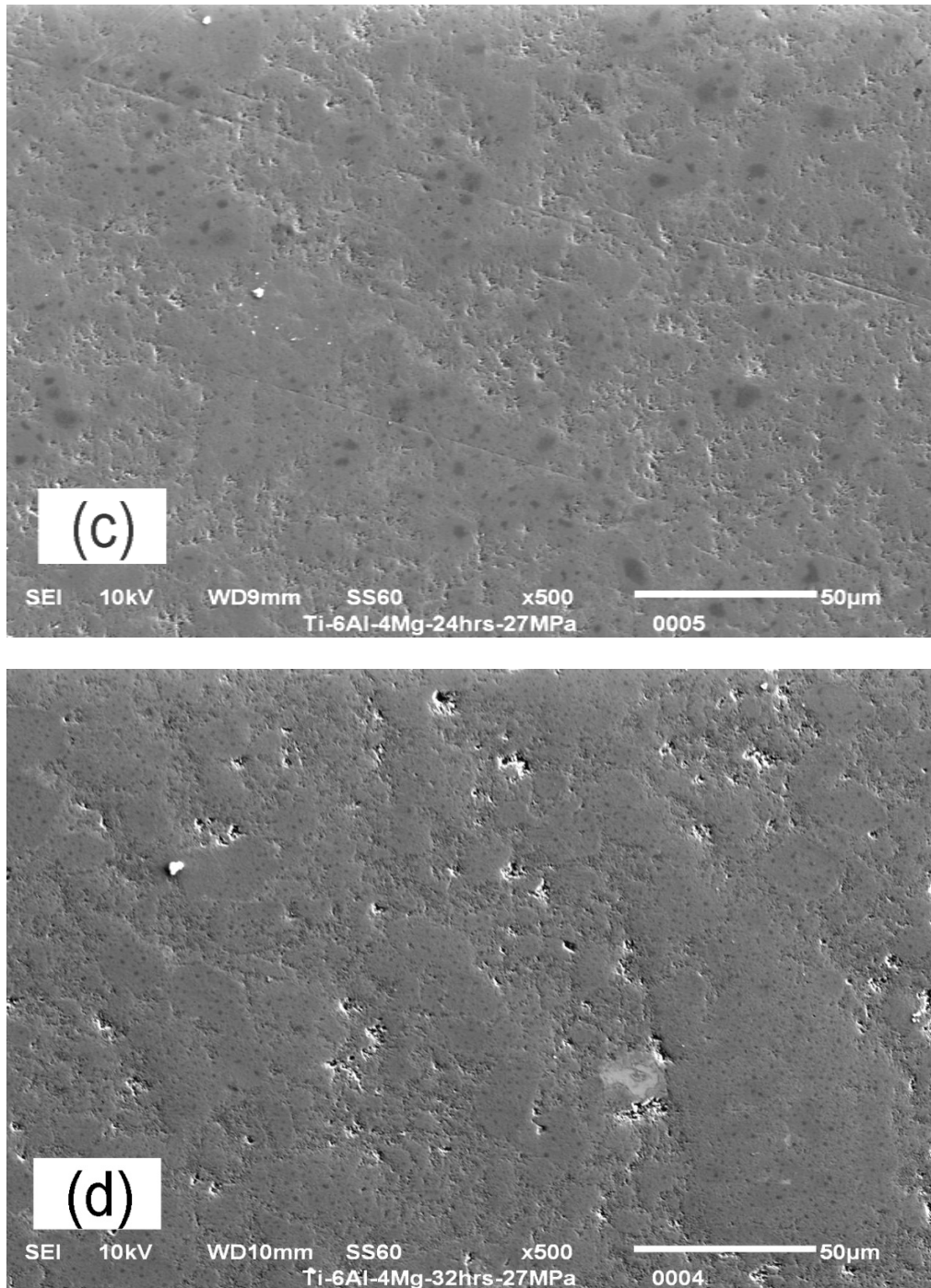


Figure 4.43: Secondary electron (SE) images of Ti-6Al-4Mg specimens pressed at 27 MPa from powders milled for (a) 24 h (b) 32 h, showing reduced porosity compared to Figure 4.64b.

The optical micrographs for Ti-6Al-4Mg specimens pressed at 33 MPa are shown in **Figures 4.44 - 4.45** while **Figures 4.46-4.47** are the corresponding SEM secondary electron images (Some back-scattered images are in **Appendix D**). The sample from powders milled for 20 hours (**Figures 4.44 b** and **4.46b**) were more porous compared to samples from the other milling times. The higher compaction pressure reduced the level of porosity in the specimens. Relatively low hardness values can be expected on the Ti-6Al-4Mg_20h_33MPa sample.

Generally, hardness is not expected to vary much across the sectioned area of the other specimens since the microstructures look uniform. The higher porosity in the specimen obtained from the powder milled for 20 hours occurred because milling for 20 hours generated a lot of fine particles (**Figure 4.8**). The fine particles are difficult to press resulting in low green densities.

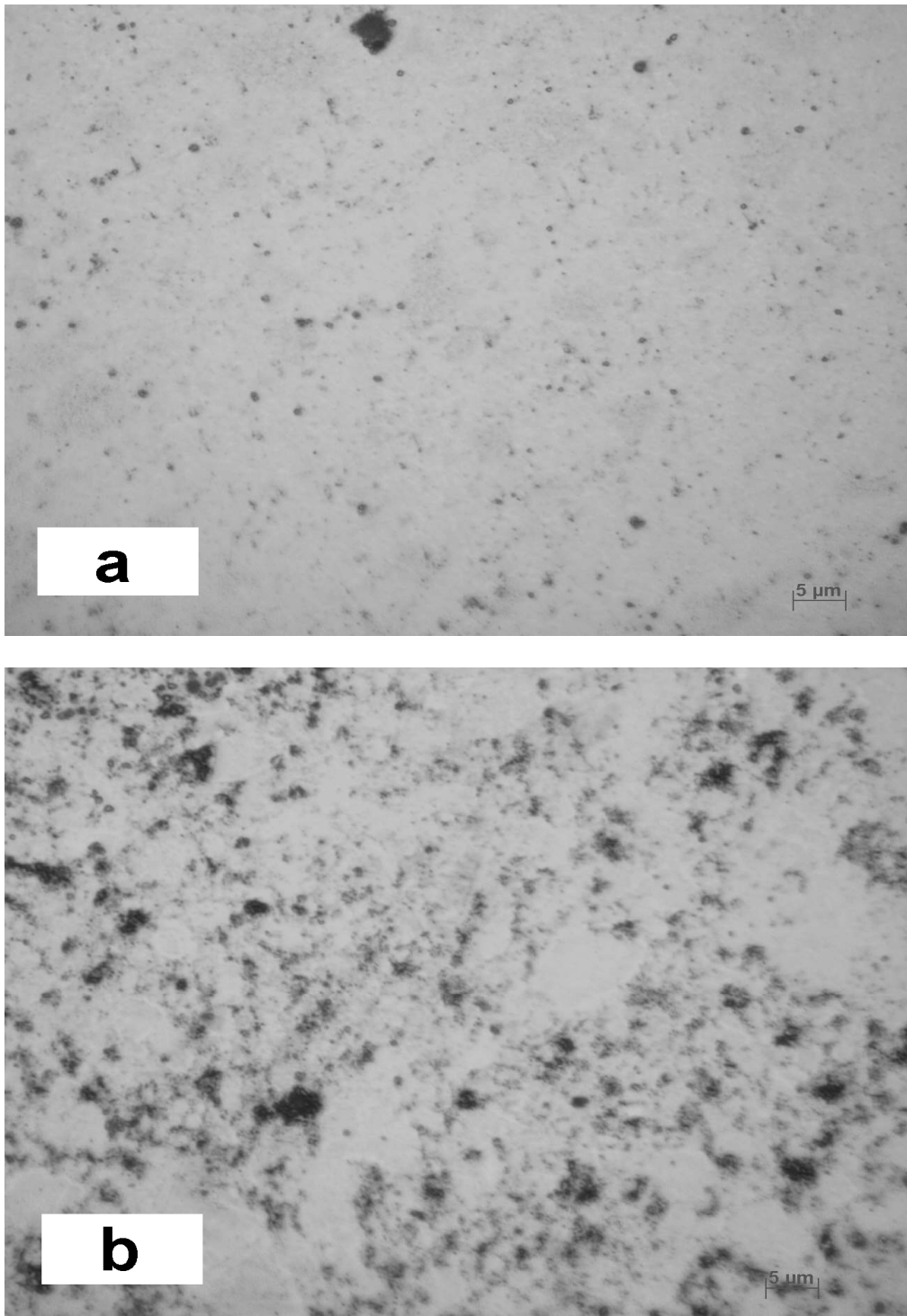


Figure 4.44: Micrographs for Ti-6Al-4Mg specimens pressed at 27 MPa from powders milled for (a) 16 h (b) 20 h.

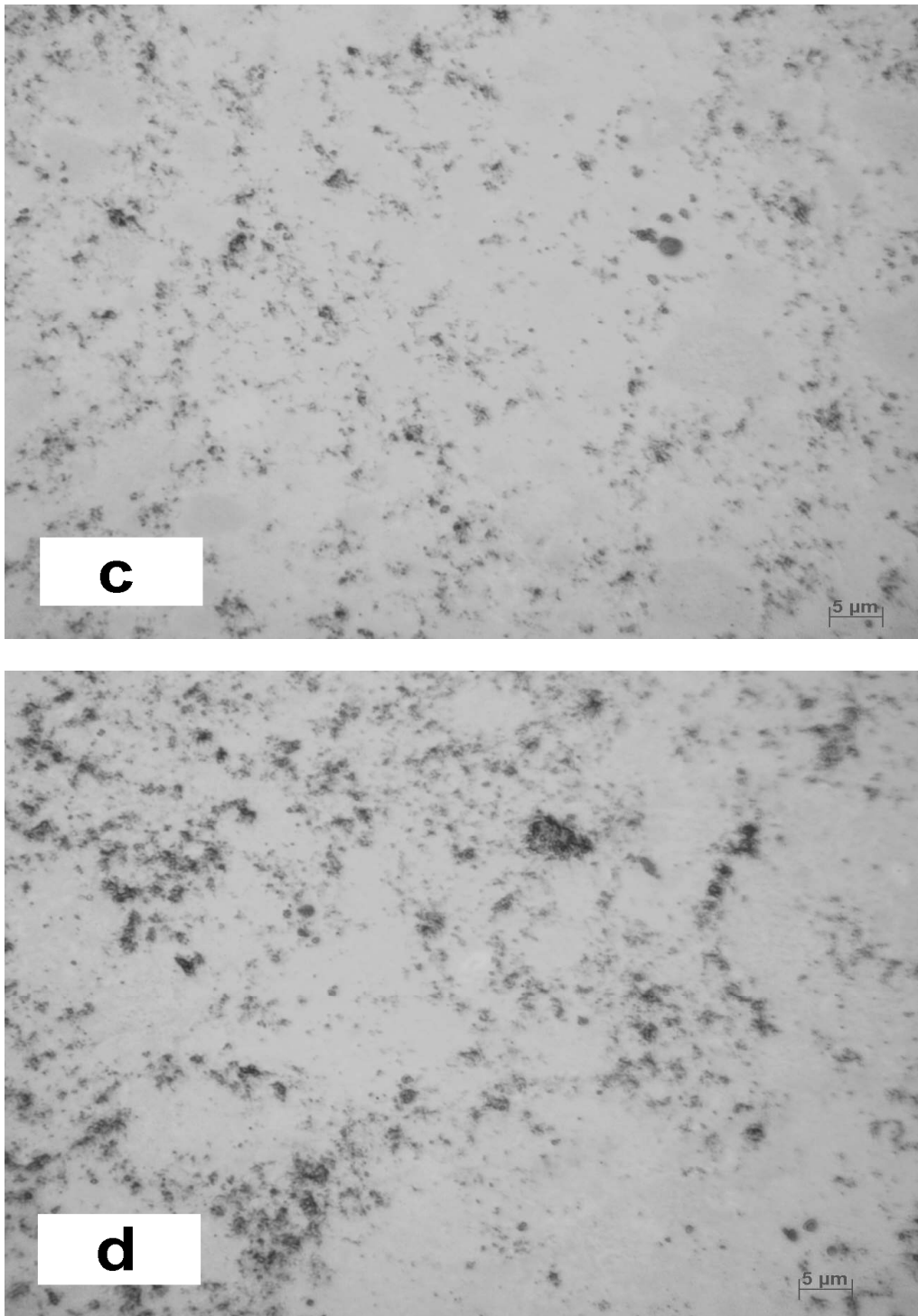


Figure 4.45: Micrographs for Ti-6Al-4Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h.

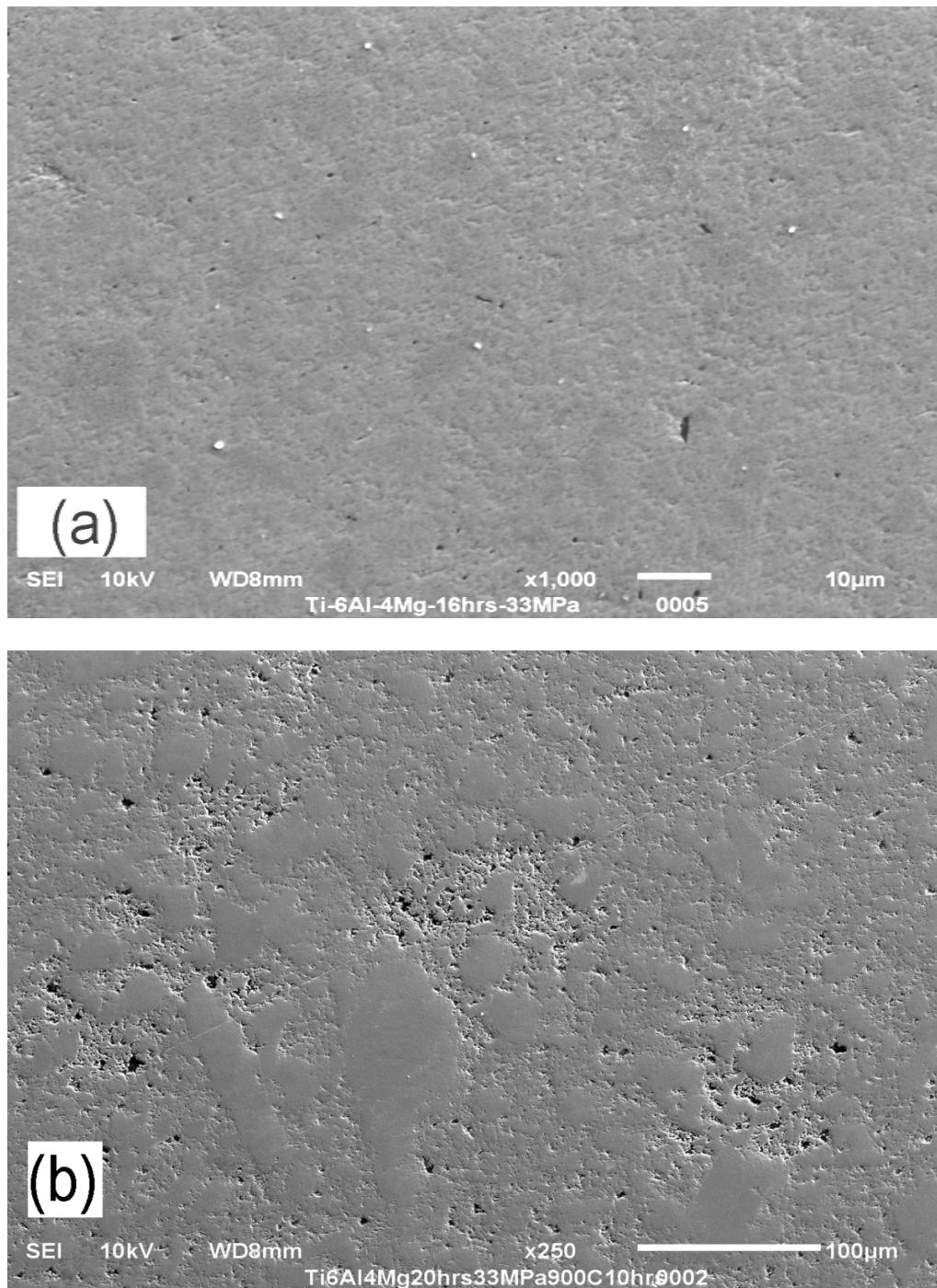


Figure 4.46: Secondary electron (SE) images of Ti-6Al-4Mg specimens pressed at 33 MPa from powders milled for (a) 16 h (b) 20 h. Relatively bigger pores can still be seen on the Ti-6Al-4Mg_20h sample.

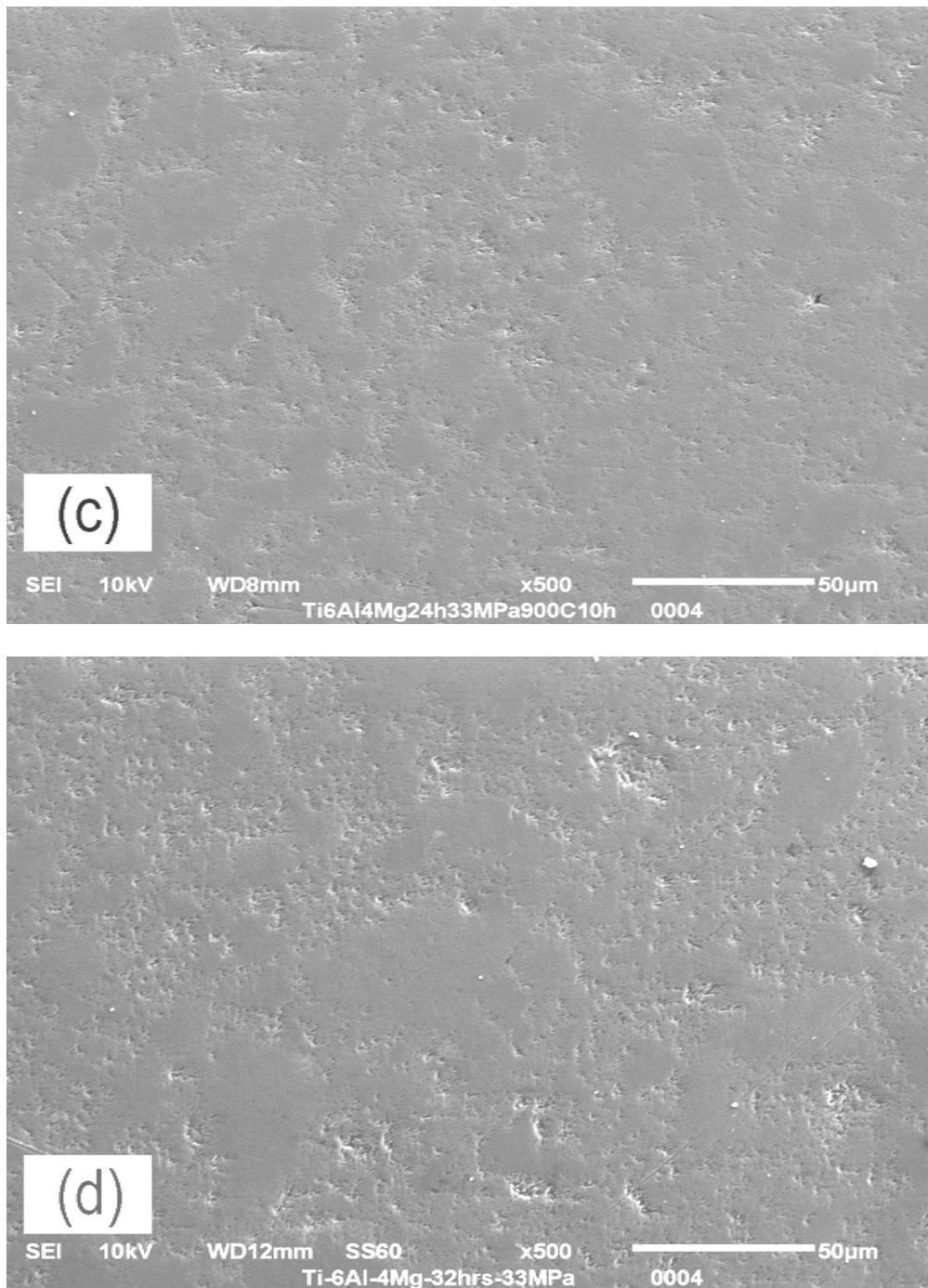


Figure 4.47: Secondary electron (SE) images for Ti-6Al-4Mg specimens pressed at 33 MPa from powders milled for (c) 24 h (d) 32 h, showing reduced porosity compared to Ti-6Al-4Mg_20h_33MPa

4.3.3 EDS of the Sintered Specimens

The chemical compositions of the sintered specimens were determined by means of Energy Dispersive Spectrometry (EDS). The EDS spectra show that all the specimens had Mg but the amount retained was always less than the nominal composition. Mg contents ranged between 0.34 and 4.13wt. %. The highest Mg contents were recorded from specimens that did not sinter properly. Approximately the same levels of carbon were measured in all the samples and it is attributed to the resin in which the samples had been mounted. Silicon from the grinding papers (during sample preparation) was detected in the most porous specimens (Ti-10Mg_20h).

4.3.3.1 Ti-10Mg specimens

The general EDS spectra revealed that the samples retained between 0.87 and 4.13 wt% of Mg (**Table 11**). The content of retained Mg was higher in samples which did not sinter. The amount of Mg retained is a promising result in the attempt to synthesize light-weight alloys. The EDS spectra for Ti-10Mg_16h_27MPa and Ti-10Mg_16h_33MPa compacts were similar to those of Ti-10Mg_32h_27MPa and Ti-10Mg_24h_33MPa. Their spectra show the presence of carbon, magnesium and titanium. The carbon was picked up from the mounting resin. **Figure 4.48** shows the EDS spectrum of Ti-10Mg_16h_27MPa.

Table 11: Chemical compositions of the sintered Ti-10Mg specimens

Specimen	Composition, wt%		
	Mg	O	Ti
Ti-10Mg_16hrs_27MPa	1.35	-	91.60
Ti-10Mg_20hrs_27MPa	3.32	-	87.78
Ti-10Mg_24hrs_27MPa	0.90	2.50	89.38
Ti-10Mg_32hrs_27MPa	1.34	-	91.63
Ti-10Mg_16hrs_33MPa	0.87	-	92.58
Ti-10Mg_20hrs_33MPa	4.13	-	66.17
Ti-10Mg_24hrs_33MPa	1.32	-	91.13
Ti-10Mg_32hrs_33MPa	1.66	2.34	88.92

However, in addition to C, Mg and Ti, Si was observed in Ti-10Mg compacts made from powders milled for 20 hours (**Figure 4.49**). The two specimens were characterised by extensive and interconnected porosity (**Figure 4.22(b)** and **4.25(b)**). Thus the silicon from the grinding papers might have collected into the pores during sample preparation.

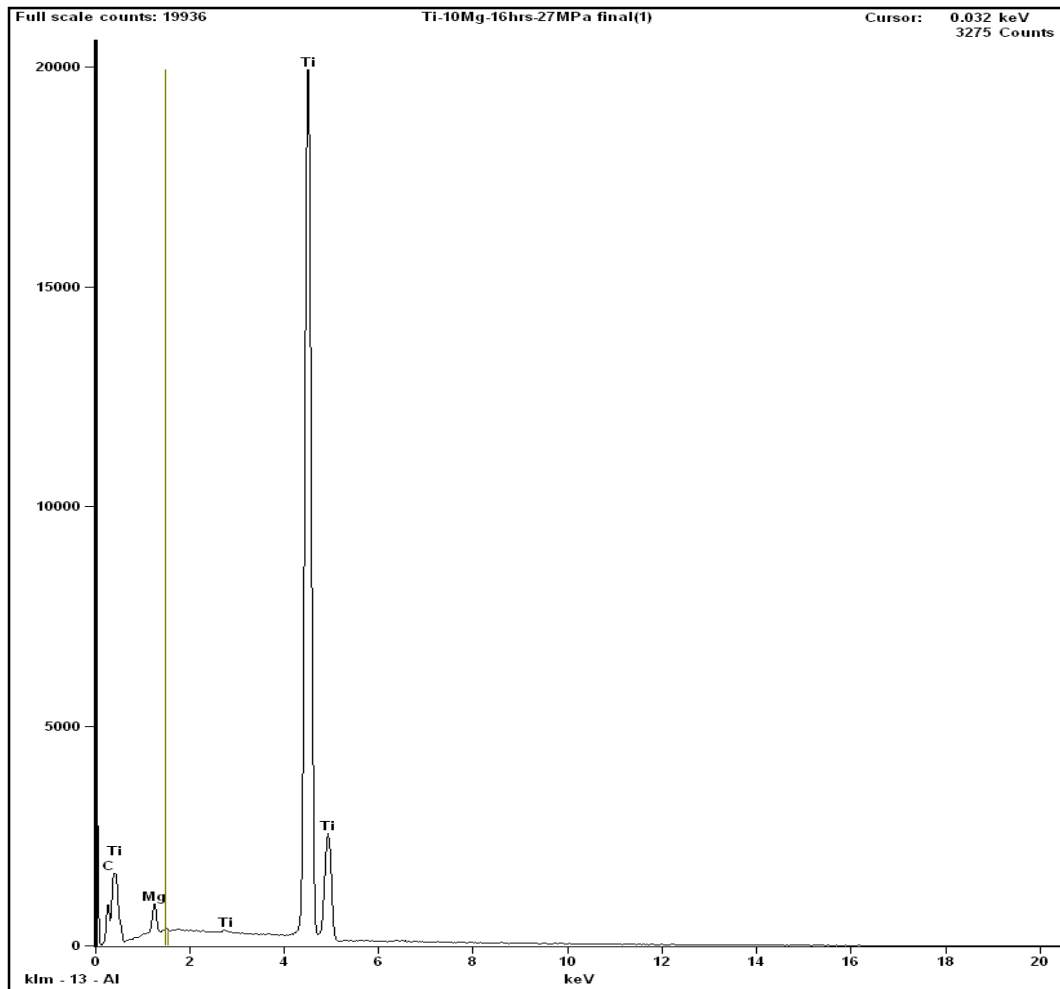


Figure 4.48: Overall EDS spectrum of the Ti-10Mg_16h_27MPa specimen

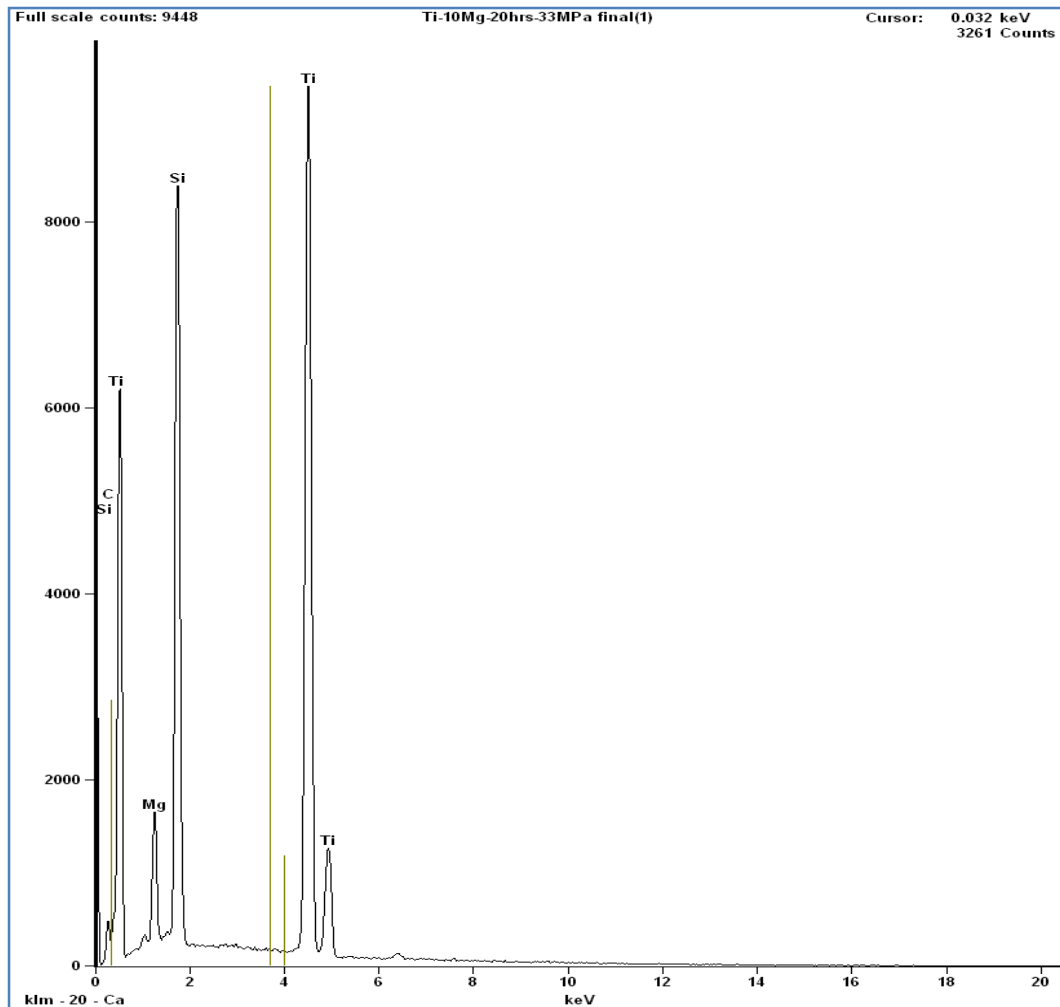


Figure 4.49: Overall EDS spectrum of the Ti-10Mg_20h_33MPa specimen showing the presence of Si from grinding papers (SiC papers)

The EDS spectrum of specimen Ti-10Mg_32h_33MPa (**Figure 4.50**) shows a peak of oxygen indicating that maybe some oxidation occurred. However, no oxides peaks appeared on the XRD pattern (**Figure 4.17**), indicating that the oxygen peak in **Figure 4.50** was due to dirt.

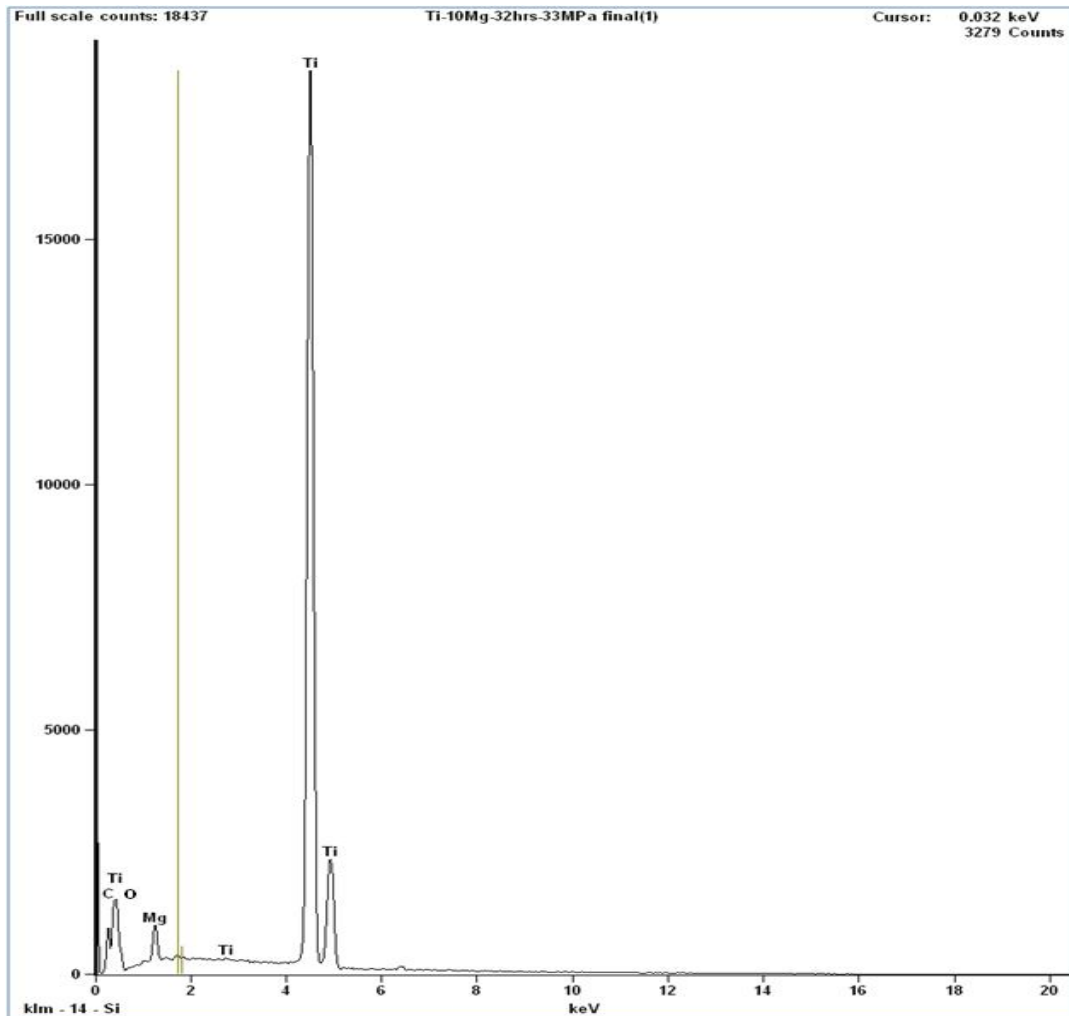


Figure 4.50: Overall EDS spectrum of the Ti-10Mg_32h_33MPa specimen showing the presence of oxygen

4.3.3.2 Ti-20Mg specimens

EDS analysis of Ti-20Mg samples showed that Mg was retained (**Figures 4.51 - 4.52**), but at much lower levels than the nominal composition. The highest Mg content obtained was 1.38wt% (for Ti-20Mg_24hrs_33MPa

sample). Compacts pressed at 27 MPa retained lower Mg compared to those pressed at 33 MPa. The Mg contents were also lower compared to Ti-10Mg specimens. Oxygen was detected for the specimens pressed at 27 MPa (from powders milled for 20, 24 and 32 hours). About 7wt% of oxygen was measured in the Ti-20Mg_20h_27MPa compared to just over 2 wt% in other samples (**Table 12**). The occurrence of oxygen here would be for the same reason as for the Ti-10Mg samples.

Table 12: Chemical compositions of the sintered Ti-20Mg specimens

Specimen	Composition, wt%		
	Mg	O	Ti
Ti-20Mg_16hrs_27MPa	0.62	-	92.04
Ti-20Mg_20hrs_27MPa	0.54	7.05	82.22
Ti-20Mg_24hrs_27MPa	0.66	2.38	90.61
Ti-20Mg_32hrs_27MPa	0.92	2.03	90.24
Ti-20Mg_16hrs_33MPa	0.95	-	91.02
Ti-20Mg_20hrs_33MPa	0.65	-	91.84
Ti-20Mg_24hrs_33MPa	1.38	-	90.90
Ti-20Mg_32hrs_33MPa	1.34	-	91.05

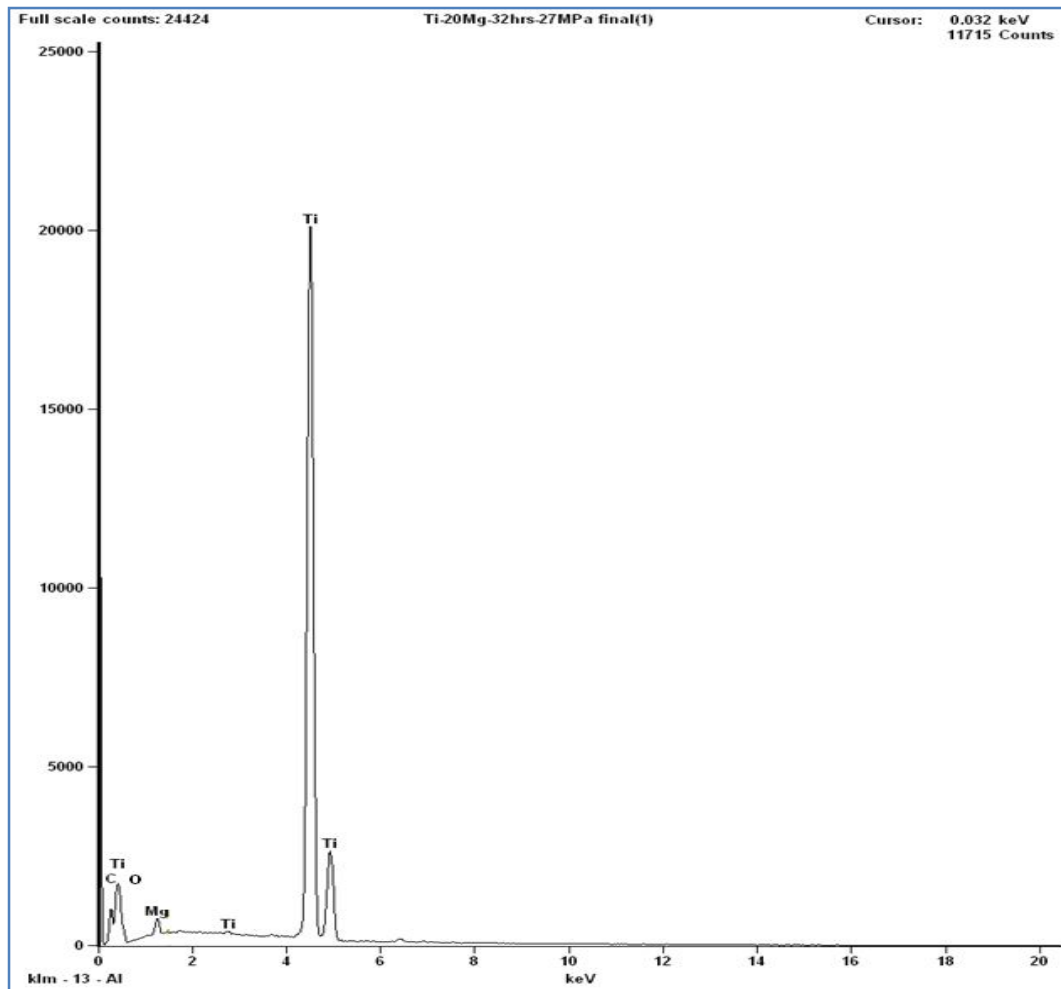


Figure 4.51: Characteristic EDS spectrum of oxygen containing samples. The pattern shown here is for Ti-20Mg_32h_27MPa.

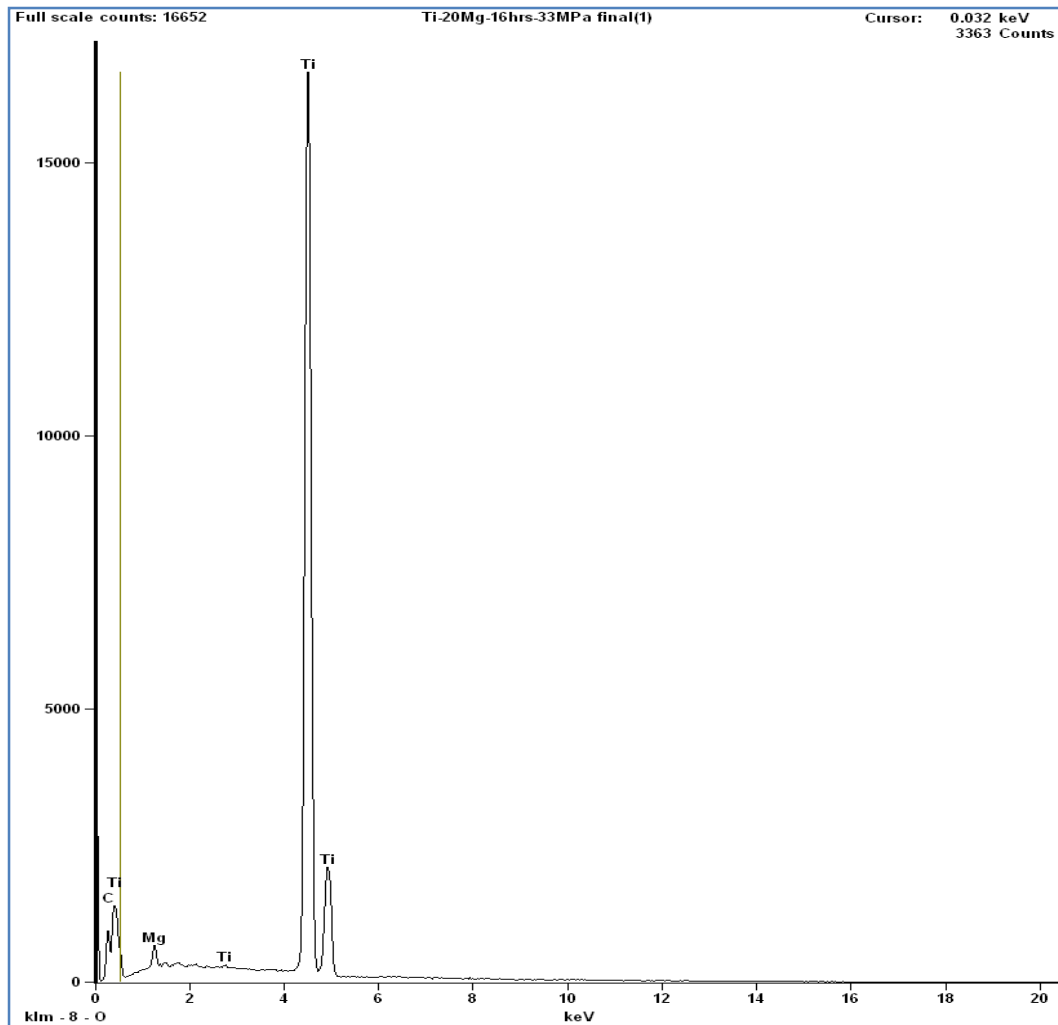


Figure 4.52: Overall EDS spectrum of the Ti-20Mg_16h_33MPa specimen

4.3.3.3 Ti-6Al-4Mg specimens

EDS analysis showed that the amount of Mg retained in Ti-6Al-4Mg samples ranged between 0.34 and 1.67 wt% (**Table 13**), better than values of 0.2wt% (0.4 at %) reported in literature [5]. At least 4 wt% of Al was retained in all the specimens (**Table 13**). Oxygen was detected in the Ti-6Al-4Mg_20h_27MPa

sample (**Figure 4.53**) only. This is expected since this sample did not sinter well and the oxygen is likely due to the accumulation of dirty in the pores. Other specimens had C, Mg, Al and Ti (**Figure 4.54**). The carbon is likely to have been a pick-up from the Bakelite resin in which the samples had been mounted.

Table 13: Chemical compositions of the sintered Ti-6Al-4Mg specimens

Specimen	Composition, wt%			
	Mg	Al	O	Ti
Ti-6Al-4Mg_16hrs_27MPa	0.63	4.80	-	86.57
Ti-6Al-4Mg_20hrs_27MPa	1.67	4.10	13.60	72.75
Ti-6Al-4Mg_24hrs_27MPa	0.83	4.91	-	87.22
Ti-6Al-4Mg_32hrs_27MPa	0.97	4.64	-	87.47
Ti-6Al-4Mg_16hrs_33MPa	0.45	4.78	-	87.11
Ti-6Al-4Mg_20hrs_33MPa	0.70	5.19	-	86.91
Ti-6Al-4Mg_24hrs_33MPa	0.34	4.91	-	90.44
Ti-6Al-4Mg_32hrs_33MPa	0.53	4.88	-	86.16

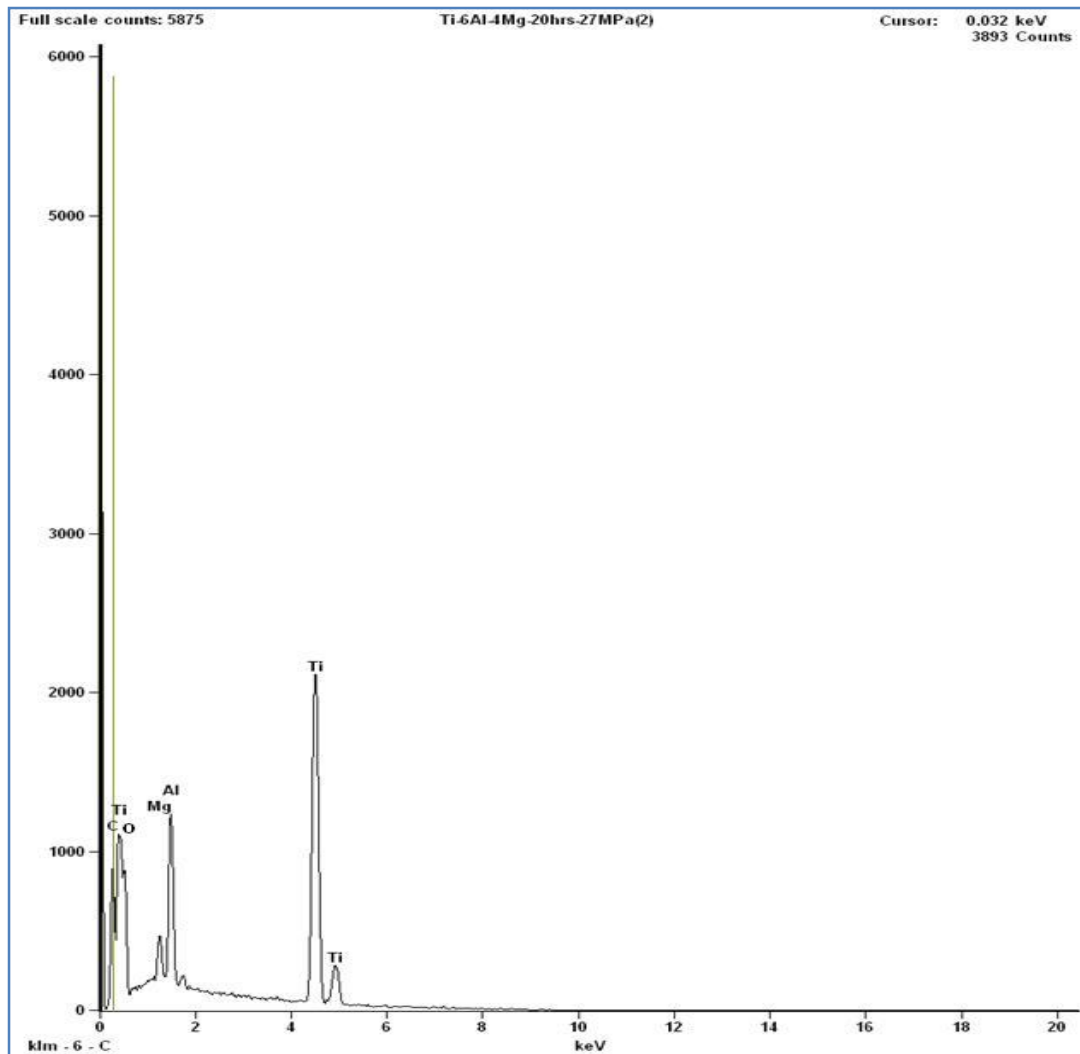


Figure 4.53: Overall EDS spectrum of the Ti-6Al-4Mg_20h_27MPa specimen showing the presence of oxygen

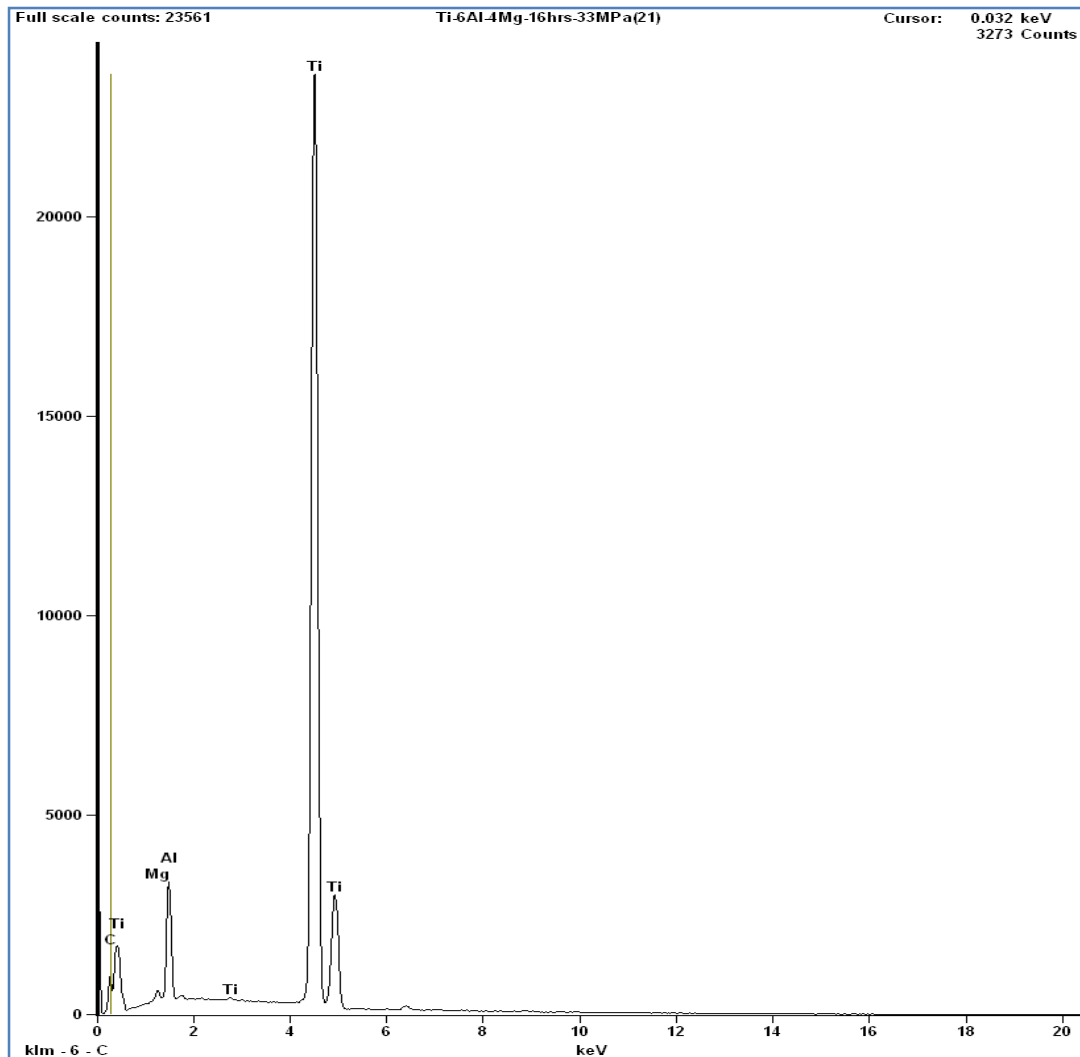


Figure 4.54: Overall EDS spectrum of the Ti-6Al-4Mg_16h_33MPa specimen

4.3.4 Hardness values

Microhardness values were observed to vary with milling time, compaction pressure and microstructure (porosity). Relatively higher values were measured on Ti-6Al-4Mg samples. Very porous specimens recorded the

lowest hardness values and these were mainly Ti-10Mg powders milled for 20 hours. Powders pressed at 33 MPa achieved higher hardness values due to reduced porosity.

4.3.4.1 Ti-10Mg hardness values

The variation of hardness values with compaction pressure and milling time for Ti-10Mg samples is given in **Figure 4.55**. The figure shows a general increase in hardness with milling time irrespective of the compaction pressure. This pattern was broken by specimens obtained from powders milled for 20 hours: these specimens had the least hardness values of 156 and 176 HV (**Appendix E**) due to their higher porosity (**Figures 4.22(b)** and **4.25(b)**).

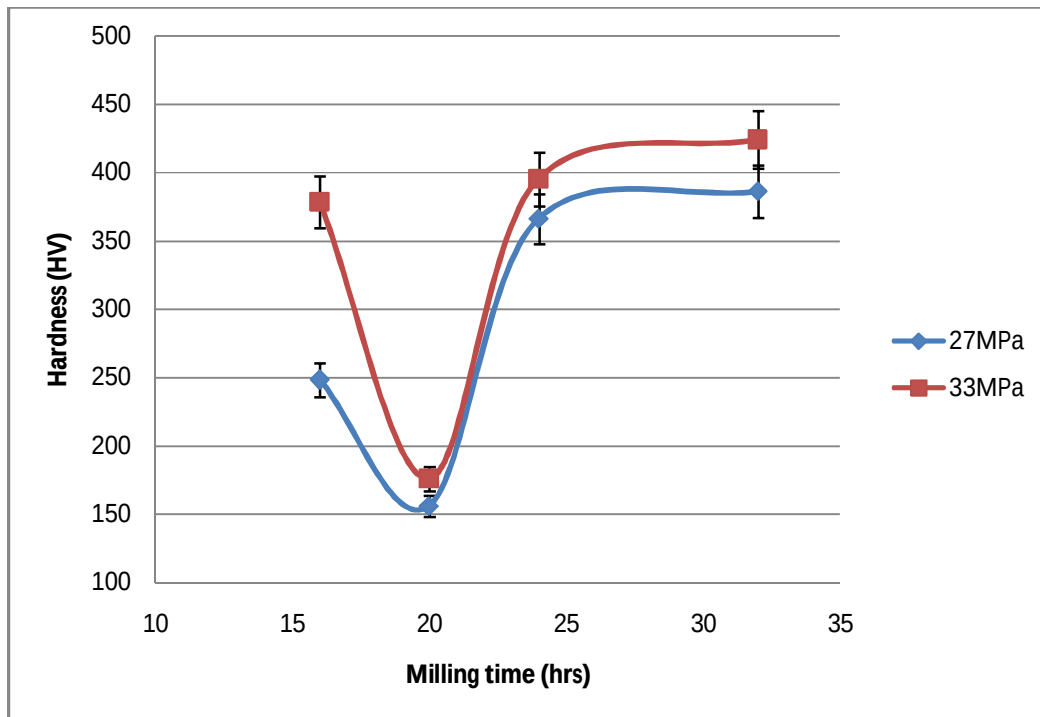


Figure 4.55: Variation of hardness values with milling time and compaction pressure for Ti-10Mg compacts sintered at 750°C

4.3.4.2 Ti-20Mg hardness values

A general increase in hardness with increasing milling time and increasing compaction pressure was observed on Ti-20Mg specimens (**Figure 4.56**). Hardness values ranged between 270 and 344 HV for compacts pressed at 27 MPa and between 307 and 427 HV for specimens obtained from powders pressed at 33MPa (**Appendix E**). The differences in hardness have been attributed to compaction load since the phase constituencies of the compacts were almost similar for each compaction pressure.

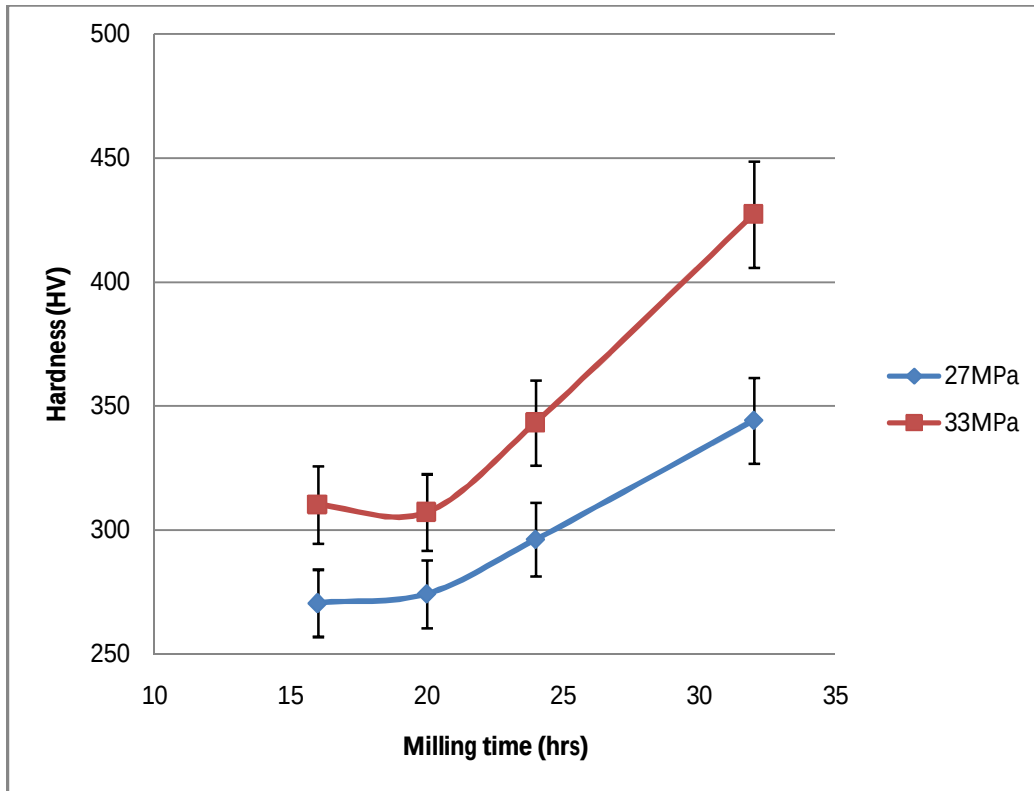


Figure 4.56: Variation of hardness values with milling time and compaction pressure for Ti-20Mg compacts sintered at 750°C

4.3.4.3 Ti-6Al-4Mg hardness values

Figure 4.57 shows the variation of hardness values with milling time for Ti-Al-4Mg specimens. Samples from powders milled for 16 hours recorded the highest hardness values of just over 910 HV (~9 GPa) (**Appendix E**), while samples obtained from powders milled for 20 hours had the least hardness for each compaction pressure. The lower hardness values of the latter samples are due to their porous structures (**Figures 4.42b** and **4.46b**). The

comparative lower porosity of the samples obtained from powders milled for 24 hours explains the increase in hardness values with increased milling. However, the hardness values do not increase beyond the values of the samples obtained from powders milled for 16 hours. It is probable that the former specimens are less hard because of the phase that formed at about 29° (2Theta) (**Figure 4.20**).

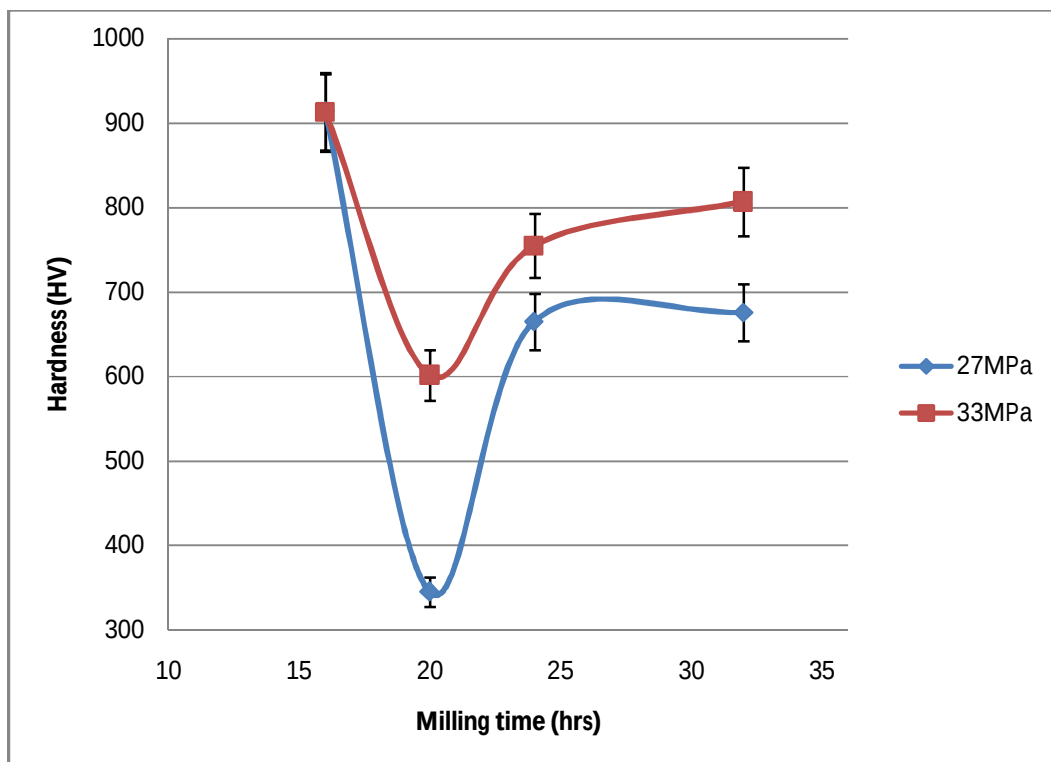


Fig 4.57: Variation of hardness values with milling time and compaction pressure for Ti-6Al-4Mg compacts sintered at 900°C

The hardness values ranging between 665 and 807 HV (**Appendix E**) obtained here for some of the Ti-6Al-4Mg samples compare well with those quoted in literature. Trindade *et al* [4] and Caetano *et al* [5] have reported hardness values of 5 GPa (510 HV) and 11 GPa (1122 HV) for Ti-5Al-10Mg and Ti-10Al-5Mg specimens, respectively. The hardness values obtained in this work are higher than those of wrought Ti-6Al-4V (~354 HV) [7].

4.3.5 Sintered Density

By measuring the sintered density of a compact, other material properties can be predicted. Sintered density can be used to monitor progress of sintering. Optimized conditions are required in order to achieve full densification. The best properties are obtained when a material has fully densified. In this work, Ti-10Mg and Ti-20Mg compacts were sintered at 630 and 750⁰C. Only the density results of the alloys sintered at 750⁰C are reported here because no sintering occurred at 630⁰C. Densities for Ti-6Al-4Mg compacts sintered at 900⁰C are also reported. Sintered compacts revealed a shiny metallic surface after sectioning. Ti-10Mg_20h_27MPa and Ti-20Mg_20h_33MPa specimens did not show that metallic finish. Observed densities higher than that of unalloyed Ti have been recorded in some sintered specimens. This might be attributed to the presence of high density Ti-oxides formed as a result of oxidation during sintering.

4.3.5.1 Sintered densities of Ti-10Mg specimens

Figure 4.58 shows the variation of sintered densities with milling time for sintered Ti-10Mg specimens. The values of density are given in **Appendix F**. Generally, specimens pressed at 33 MPa achieved relatively low sintered densities. The Ti-10Mg_20h_27MPa recorded the lowest as-sintered density. The microstructure of this sample was characterised by massive porosity (**Figure 4.22 b**)). This argument would imply that the specimen obtained from powder milled for 20 hours and pressed with 33 MPa should also have had a lower density. It is possible that the sample from 27 MPa had closed porosity while that from 33 MPa had open porosity. Further work is continuing to characterize the porosities better.

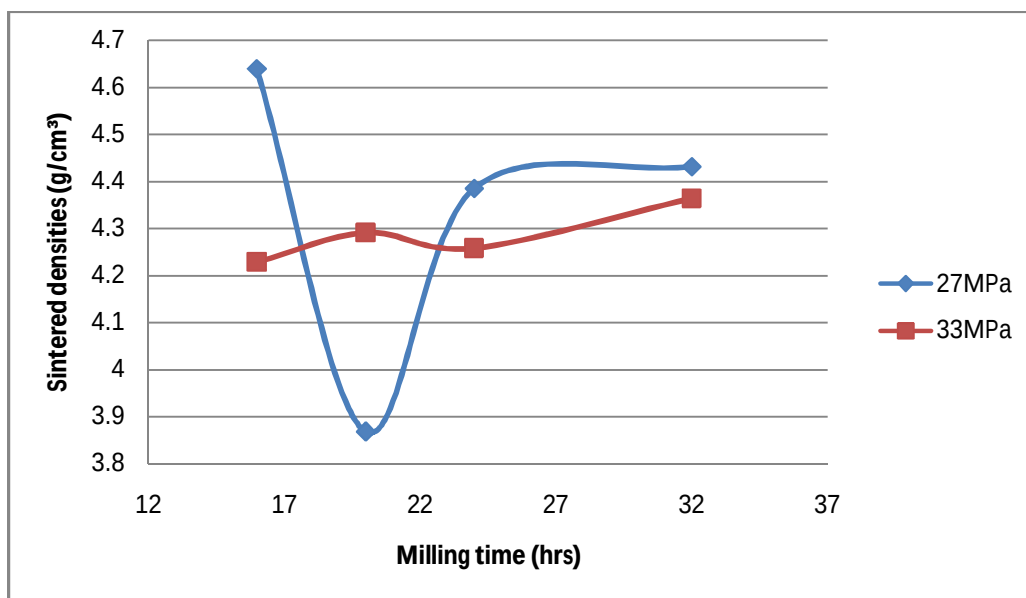


Figure 4.58: Variation of densities with milling time and compaction pressure for Ti-10Mg specimens sintered at 750°C for 5 hours.

The density differences between the samples from powders milled for 16 hours could be due to the difference in phase composition (**Figures 4.16a** and **4.17a**). The specimen pressed at 27 MPa has one more phase than the specimen pressed at 33 MPa.

4.3.5.2 Sintered densities of Ti-20Mg specimens

Figure 4.59 shows the variation of sintered densities with milling time for sintered Ti-20Mg specimens. The raw data is presented in **Appendix F**. Generally, the sintered densities decreased with increasing milling time. This was clearly the case for samples produced from pressing at 27 MPa. The density differences between the samples pressed at 27 and 33 MPa for samples obtained from powders milled for 24 and 32 hours were insignificant. This is due to the low variation in the particle size distribution of the milled powders (**Table 7**). Also, the specimens had similar phases.

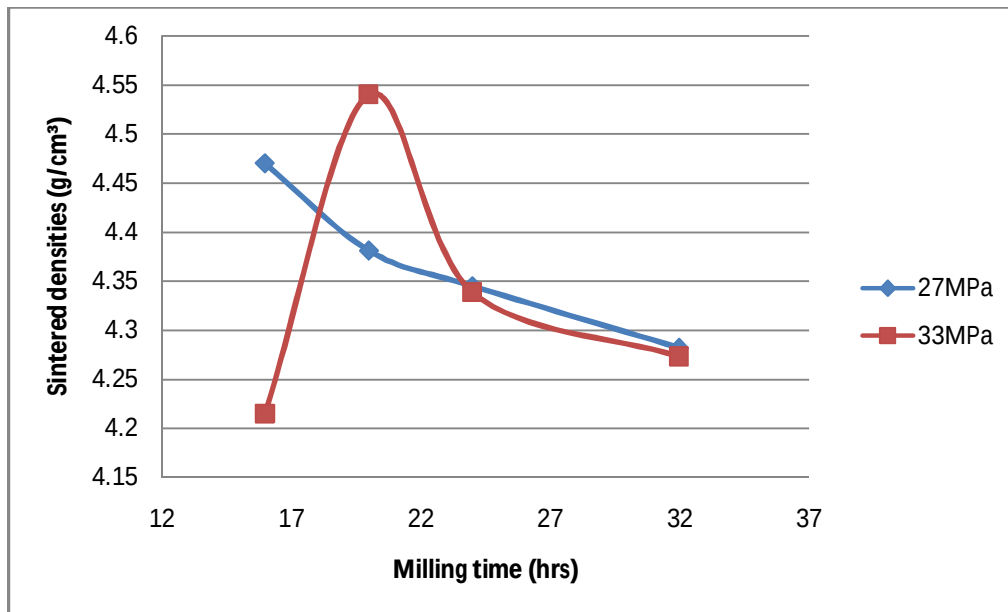


Figure 4.59: Variation of densities with milling time and compaction pressure for Ti-20Mg specimens sintered at 750°C for 5 hours.

4.3.5.3 Sintered densities of Ti-6Al-4Mg specimens

Figure 4.60 shows the variation of density of sintered Ti-6Al-4Mg compacts with milling time and compaction pressure. The values of the densities are given in **Appendix F**. Samples from powder milled for 16 hours have the same density, and the variation of density, observed as decreasing with milling time, for powders milled for 24 and 32 hours is similar.

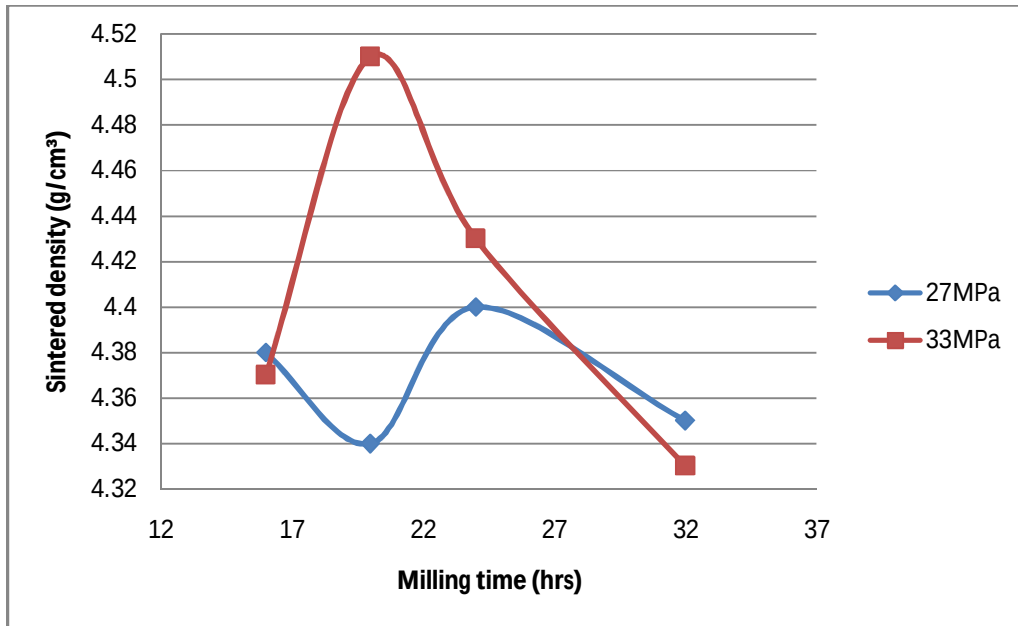


Figure 4.60: Variation of densities with milling time for Ti-6Al-4Mg specimens sintered at 900C for 10 hours.

The variation of density for the samples of compacts pressed at 27 MPa is similar to the Ti-10Mg samples obtained from the same load (**Figure 4.58**). The same can be said for specimens obtained from powder pressed with 33 MPa which mirrors that of Ti-20Mg at the same compaction pressure (**Figure 4.59**). The very density of the sample obtained from powder milled for 20 hours and compacted at 33 MPa is possibly due to open porosity that allowed pore penetration. This could have led to a smaller volume being determined.

CHAPTER FIVE

SUMMARY OF RESULTS AND CONCLUSION

This study set out to sinter Ti-Mg and Ti-6Al-4Mg alloy powders obtained from the high energy milling of elemental powders. The Ti-Mg and Ti-6Al-4Mg compacts were successfully sintered at 750 and 900°C respectively. At 630°C, all the specimens did not sinter. Particle size distribution and compaction pressure affected both green and sintered properties.

Particle size analysis revealed that Ti-10Mg and Ti-6Al-4Mg powders milled for 20 hours had more fine particles compared to those from other milling times (**Figures 4.1 and 4.8**). Further milling led to an increase in particle size for all powders. This increase can be attributed to cold-welding dominating fracturing or to the fine particles agglomerating. This agglomeration was observed in the morphological studies of the milled powders. After milling, it was found that the granular and irregularly shaped particles that formed were agglomerates of smaller particles.

The characterization of the green compacts has shown that the green densities appeared to decrease with increasing milling time (**Figures 4.11-4.13**). Relatively lower green densities were recorded on powders milled for 20 hours. As has already been noted above, these powders had a lot of fine

particles. Fine particles are difficult to press because they possess relatively higher surface area generating higher inter-particle friction that results in a decrease of the effective force. A higher compaction pressure led, generally, to higher green densities for all the powders (**Figures 4.11-4.13**). This is because higher pressures result in increased particle-contact enlargement as a result of particles rearranging and sliding over each other. Higher pressures also encourage deformation and cold-welding between particles, hence increase in density.

XRD spectra of the sintered samples had sharp, narrow peaks (**Figures 4.16 – 4.21**) compared to those of the milled powders (**Figures 3.1 – 3.3**). This indicates that the size of the particles interacting with X-rays had become bigger and relatively strain-free due to the lower amount of Mg in solution. The amount of retained Mg in the sintered samples, obtained by EDS analysis, ranged between 0.34 and 4.13wt% (**Tables 11 – 13**), much less than the nominal compositions (**Tables 1 – 3**). The loss of Mg can be explained by the fact that the high Mg-content solid solutions of Ti(Mg) and Ti(Al,Mg) formed in the powders are metastable, and would dissociate to the equilibrium phases of Ti and Mg when subjected to high enough temperatures. The equilibrium solubility of Mg in Ti is very low even at high temperatures [20]. It appears therefore that the method of consolidation used in this study was not well suited to the alloys.

The dissociation of the Ti(Mg) solid solutions formed in the powders lead to the formation of peaks of mostly elemental Ti (**Figures 4.16 – 4.19**). No extra peaks were obtained for Ti-10Mg samples obtained from powder milled for 20 hours. This might have occurred because the extent of sintering of these latter samples was very low (**Figure 4.22 b**) and **4.25 b**). The dissociation of the Ti(Mg) solid solutions observed here is in line with literature [5], and shows that the consolidation process used was probably incompatible with the powders. XRD analysis also revealed the formation of titanium oxides. Higher compaction pressure and finer particle size distribution seem to have favoured formation of these oxides. Sintering at 900°C also resulted in more specimens being oxidised compared to a temperature of 750°C.

The sintering behaviour of the Ti-6Al-4Mg powders was similar to that of the Ti-Mg powders, even though apart from the dissociation of the Ti(Al,Mg) solid solutions formed in the powders, there was also some oxidation. The oxidation occurred even though the sintering was done under argon. It is possible that the argon used was contaminated with oxygen. Thus, sintering in vacuum might minimise oxidation.

The microstructures of all the sintered samples were porous. The porosity was especially pronounced for Ti-10Mg and Ti-6Al-4Mg samples obtained from powder milled for 20 hours (See e.g. **Figures 4.22 b**), **4.25 b**), **4.27 b**) (for Ti-10Mg) and **4.40 b**), **4.42** and **4.46** (for Ti-6Al-4Mg) in Section 4.3.2.).

The extensive porosity in these samples was caused by the fact that the green densities were, as has already been mentioned, low due to the high amounts of fines in the powders. Overall, the occurrence of porosity in the microstructures of the sintered specimens could, again, be an indication that the pressureless sintering employed in this study was insufficient to achieve full densification. Porosity could also be a result of the presence of unsintered powder particles in specimens after sintering. Compacts pressed at 33 MPa achieved relatively lower porosities. Ti-6Al-4Mg compacts sintered at 900°C were less porous compared to their respective Ti-10Mg and Ti-20Mg compacts sintered at 750°C. Therefore, temperature has an influence on porosity.

The hardness values of the sintered samples generally appeared to increase with milling time for Ti-10Mg and Ti-20Mg samples (**Figures 4.55** and **4.56**). The lowest hardness values of 156.0 and 176.0 HV for Ti-10Mg samples were obtained for samples from powders milled for 20 hours. This has been attributed to the porosity of the specimens. The hardness values steadily increased with increasing milling time for Ti-20Mg specimens irrespective of the compaction pressure. Hardness ranged between 270.8 and 427.4 HV. This trend could have been assisted by the fact that the phases formed during sintering of Ti-20Mg compacts were similar, and also the decrease in porosity of specimens with increasing milling time. The variation of hardness with milling time for Ti-6Al-4Mg appears to have been affected by both phase

composition and porosity. The hardness was higher (**Figure 4.57**) when there was no oxidation as in the case of Ti-6Al-4Mg_16hrs_27MPa (**Figure 4.20**) and lower when porosity was high as in the case of Ti-6Al-4Mg-20hrs-27MPa (**Figure 4.40(b)**). Hardness values of up to 914 HV were measured in these samples. Compaction pressure affected hardness with higher pressures producing relatively higher values.

The sintered density of Ti-10Mg samples generally increased with milling time (**Figure 4.58**). This trend was different from those of Ti-20Mg and Ti-6Al-4Mg samples for which density generally decreased with milling time (**Figures 4.59** and **4.60**). The trend in the latter two alloys is line with the behaviour of the green densities (**Figures 4.11-4.13**). It is possible that the different variations of the densities were caused by differences in the morphologies of the porosities of the different sintered samples. No attempts were made to characterize the porosities. Densities as low as 4.23 and 4.21 g/cm³ were achieved in Ti-10Mg and Ti-20Mg specimens respectively while Ti-6Al-4Mg sample had a density of 4.33 g/cm³. These density results are promising since they are comparatively lower than those of most Ti-based alloys.

The results show that particle size distribution of the milled powders has an effect on both green and sintered body properties. Powders milled for 20 hours had a lot of fine particles irrespective of the alloy composition. It has been observed that a large amount of fines (less than 1µm) are not desirable

as they lead to low green strengths and porous microstructures. These porous microstructures negatively affected hardness and other mechanical properties such as strength. Higher compaction pressures assisted in achieving less porous specimens. A temperature of 630°C was observed to be too low for sintering. Sintering was successful at 750 and 900°C. Higher sintering temperatures (and compaction pressure) favoured oxidation and low porosity. Losses in Mg were significant which suggest that these temperatures employed might be too high or the sintering times were longer. Thus, methods such as Spark Plasma Sintering, which allow sintering to be carried out at the shortest possible time, can be effective in reducing Mg losses. The low densities achieved on some of the less porous specimens are a promising result in the attempt to produce low-density Ti-Mg based alloys. There could also be need to change from argon to vacuum in order to minimise oxidation.

CHAPTER SIX

RECOMMENDATIONS

The following recommendations are being made in order to address some of the issues that have been highlighted in this study:

6.1 Porosity:

The porosity observed in the microstructures of the samples obtained in this study could have been caused by the levels of the compaction pressures used to produce the green compacts. It is being recommended that higher pressures be used to achieve higher green densities, which in turn gives less porous specimens.

The green densities were affected negatively by powder particle size distribution. Higher amounts of fines were generated by milling for 20 hours. It is being recommended here that future work can avoid milling for this duration in order to avoid the formation of fines.

6.2 Oxidation:

The oxidation observed for some of the samples might have been caused by contamination of the inert gas used. This can be avoided by sintering under vacuum. Sintering at temperatures less than 900°C might also minimise oxidation.

6.3 Loss of magnesium

The loss of magnesium experienced for all the samples in this study emanated from the dissociation of the Ti(Mg) and Ti(Al,Mg) solid solutions formed in the powders on milling. These solid solutions are metastable phases and it would be expected that they would tend to their equilibrium states under temperature and time conditions that allow diffusion to occur. The dissociation can however be stopped if the sintering time can be shortened. It is being recommended that newer sintering techniques, like spark plasma sintering (SPS), be used in future to sinter the powders.

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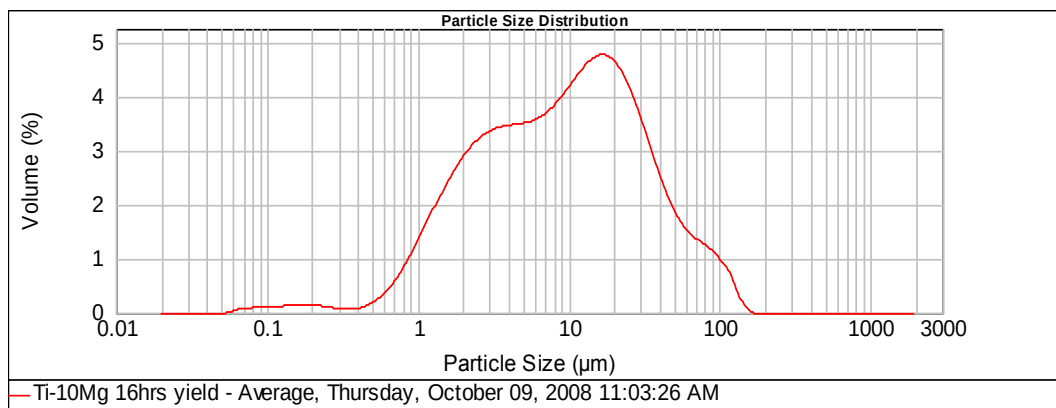
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APPENDICES

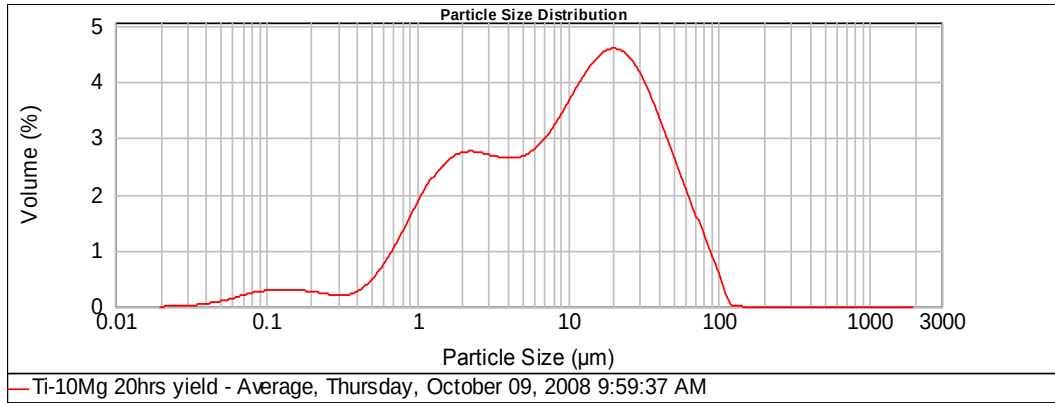
Appendix A: Raw Data and Particle Size Distribution Curves

Ti-10Mg_16hrs Yield



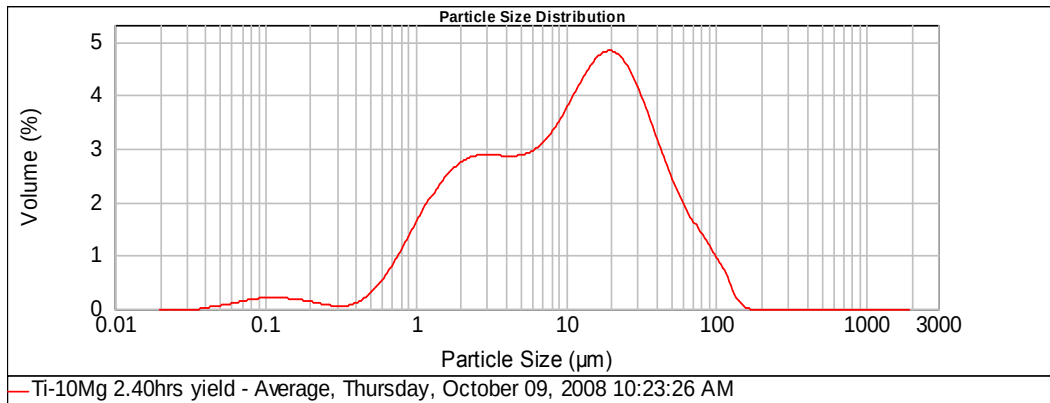
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0.010	0.00	0.080	0.15	0.634	1.98	5.053	34.38	40.244	89.02	320.535	100.00
0.011	0.00	0.090	0.23	0.717	2.39	5.709	37.19	45.469	90.91	362.148	100.00
0.013	0.00	0.102	0.32	0.810	2.96	6.450	40.04	51.371	92.51	409.163	100.00
0.014	0.00	0.115	0.42	0.915	3.73	7.287	42.96	58.041	93.89	462.281	100.00
0.016	0.00	0.130	0.52	1.034	4.72	8.233	45.98	65.575	95.11	522.296	100.00
0.018	0.00	0.147	0.62	1.168	5.96	9.302	49.14	74.089	96.21	590.102	100.00
0.021	0.00	0.166	0.73	1.320	7.45	10.510	52.45	83.707	97.22	666.711	100.00
0.024	0.00	0.187	0.85	1.491	9.17	11.874	55.92	94.574	98.13	753.265	100.00
0.027	0.00	0.211	0.96	1.684	11.13	13.416	59.54	106.852	98.91	851.056	100.00
0.030	0.00	0.239	1.06	1.903	13.28	15.157	63.28	120.724	99.54	961.542	100.00
0.034	0.00	0.270	1.15	2.150	15.62	17.125	67.08	136.397	99.87	1086.372	100.00
0.038	0.00	0.305	1.22	2.429	18.10	19.348	70.86	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	1.28	2.745	20.69	21.860	74.55	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	1.34	3.101	23.35	24.698	78.05	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	1.41	3.503	26.07	27.904	81.30	222.251	100.00	1770.189	100.00
0.062	0.02	0.497	1.52	3.958	28.83	31.527	84.23	251.105	100.00	2000.000	100.00
0.070	0.08	0.561	1.70	4.472	31.60	35.620	86.80	283.704	100.00		

Ti-10Mg_20hrs Yield



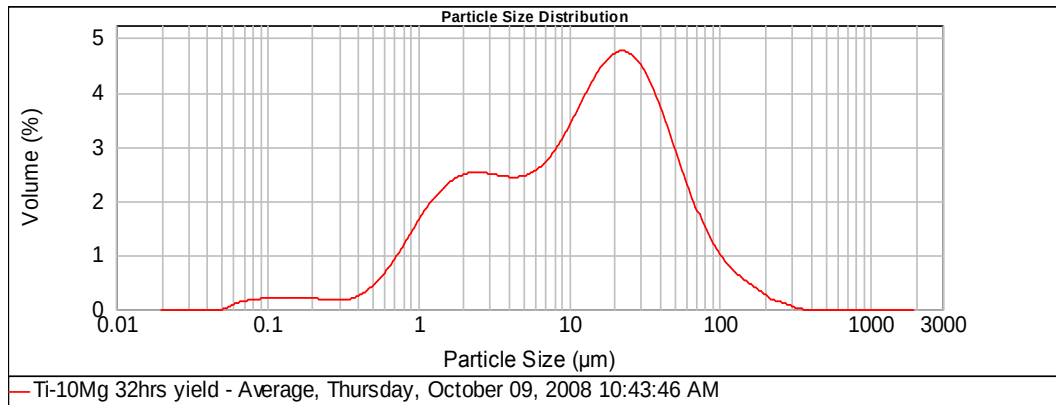
Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	0.080	0.68	0.634	4.87	5.053	35.93	40.244	87.87	320.535	100.00
0.011	0.00	0.090	0.89	0.717	5.62	5.709	38.09	45.469	90.44	362.148	100.00
0.013	0.00	0.102	1.11	0.810	6.57	6.450	40.34	51.371	92.70	409.163	100.00
0.014	0.00	0.115	1.34	0.915	7.74	7.287	42.69	58.041	94.66	462.281	100.00
0.016	0.00	0.130	1.58	1.034	9.13	8.233	45.19	65.575	96.32	522.296	100.00
0.018	0.00	0.147	1.81	1.168	10.74	9.302	47.87	74.089	97.66	590.102	100.00
0.021	0.00	0.166	2.04	1.320	12.53	10.510	50.74	83.707	98.71	666.711	100.00
0.024	0.00	0.187	2.27	1.491	14.49	11.874	53.81	94.574	99.44	753.265	100.00
0.027	0.00	0.211	2.48	1.684	16.56	13.416	57.07	106.852	99.89	851.056	100.00
0.030	0.01	0.239	2.68	1.903	18.71	15.157	60.49	120.724	99.99	961.542	100.00
0.034	0.03	0.270	2.86	2.150	20.91	17.125	64.05	136.397	100.00	1086.372	100.00
0.038	0.06	0.305	3.01	2.429	23.11	19.348	67.70	154.104	100.00	1227.408	100.00
0.043	0.10	0.345	3.17	2.745	25.30	21.860	71.36	174.110	100.00	1386.753	100.00
0.049	0.17	0.389	3.34	3.101	27.46	24.698	74.99	196.714	100.00	1566.785	100.00
0.055	0.25	0.440	3.56	3.503	29.60	27.904	78.51	222.251	100.00	1770.189	100.00
0.062	0.36	0.497	3.87	3.958	31.71	31.527	81.87	251.105	100.00	2000.000	100.00
0.070	0.51	0.561	4.30	4.472	33.81	35.620	85.00	283.704	100.00		

Ti-10Mg_24hrs Yield



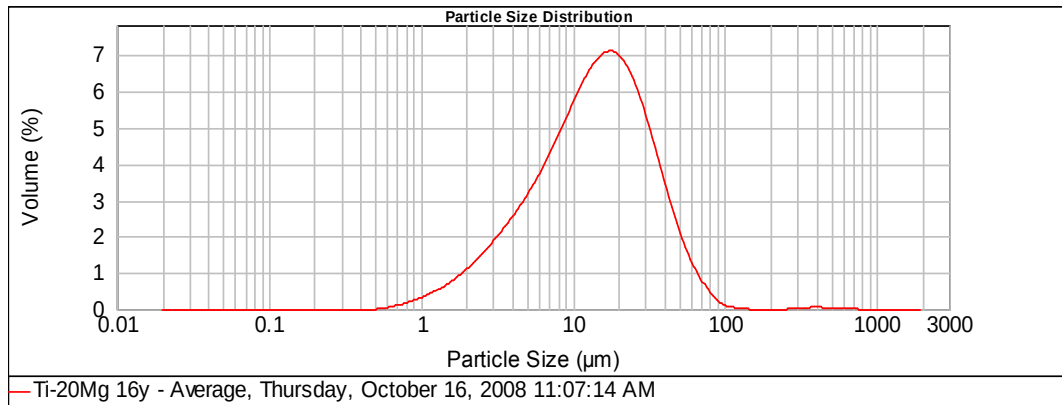
Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	0.080	0.42	0.634	2.85	5.053	33.50	40.244	87.02	320.535	100.00
0.011	0.00	0.090	0.57	0.717	3.42	5.709	35.81	45.469	89.44	362.148	100.00
0.013	0.00	0.102	0.73	0.810	4.19	6.450	38.18	51.371	91.54	409.163	100.00
0.014	0.00	0.115	0.90	0.915	5.17	7.287	40.63	58.041	93.35	462.281	100.00
0.016	0.00	0.130	1.06	1.034	6.37	8.233	43.21	65.575	94.91	522.296	100.00
0.018	0.00	0.147	1.22	1.168	7.79	9.302	45.96	74.089	96.24	590.102	100.00
0.021	0.00	0.166	1.37	1.320	9.42	10.510	48.91	83.707	97.38	666.711	100.00
0.024	0.00	0.187	1.50	1.491	11.24	11.874	52.08	94.574	98.33	753.265	100.00
0.027	0.00	0.211	1.61	1.684	13.22	13.416	55.47	106.852	99.09	851.056	100.00
0.030	0.00	0.239	1.70	1.903	15.32	15.157	59.05	120.724	99.65	961.542	100.00
0.034	0.00	0.270	1.76	2.150	17.52	17.125	62.79	136.397	99.91	1086.372	100.00
0.038	0.00	0.305	1.80	2.429	19.78	19.348	66.63	154.104	100.00	1227.408	100.00
0.043	0.02	0.345	1.84	2.745	22.06	21.860	70.47	174.110	100.00	1386.753	100.00
0.049	0.05	0.389	1.89	3.101	24.36	24.698	74.24	196.714	100.00	1566.785	100.00
0.055	0.11	0.440	1.99	3.503	26.66	27.904	77.84	222.251	100.00	1770.189	100.00
0.062	0.19	0.497	2.16	3.958	28.94	31.527	81.20	251.105	100.00	2000.000	100.00
0.070	0.29	0.561	2.43	4.472	31.22	35.620	84.27	283.704	100.00		

Ti-10Mg_32hrs Yield



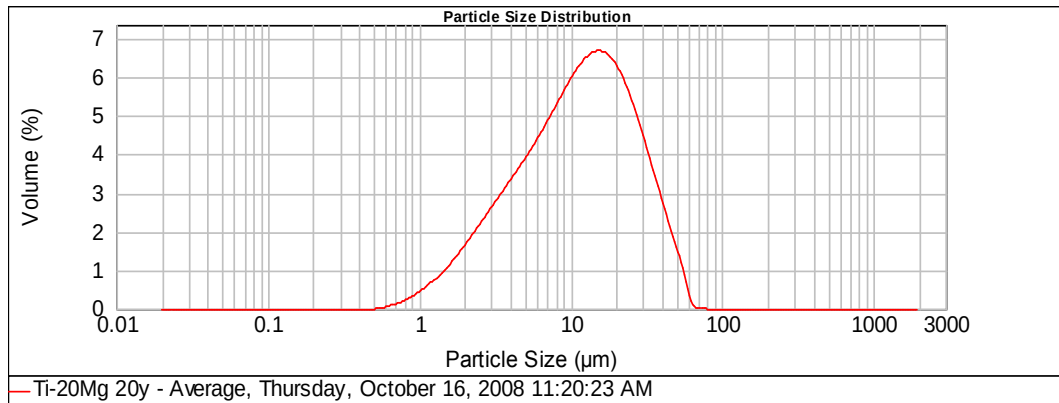
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
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0.011	0.00	0.090	0.44	0.717	4.36	5.709	33.80	45.469	86.31	362.148	100.00
0.013	0.00	0.102	0.60	0.810	5.20	6.450	35.84	51.371	88.83	409.163	100.00
0.014	0.00	0.115	0.77	0.915	6.23	7.287	37.98	58.041	91.00	462.281	100.00
0.016	0.00	0.130	0.94	1.034	7.46	8.233	40.26	65.575	92.83	522.296	100.00
0.018	0.00	0.147	1.11	1.168	8.88	9.302	42.71	74.089	94.34	590.102	100.00
0.021	0.00	0.166	1.28	1.320	10.47	10.510	45.37	83.707	95.57	666.711	100.00
0.024	0.00	0.187	1.44	1.491	12.21	11.874	48.25	94.574	96.56	753.265	100.00
0.027	0.00	0.211	1.60	1.684	14.06	13.416	51.37	106.852	97.35	851.056	100.00
0.030	0.00	0.239	1.76	1.903	16.00	15.157	54.71	120.724	97.99	961.542	100.00
0.034	0.00	0.270	1.90	2.150	17.99	17.125	58.25	136.397	98.52	1086.372	100.00
0.038	0.00	0.305	2.02	2.429	19.99	19.348	61.93	154.104	98.95	1227.408	100.00
0.043	0.00	0.345	2.16	2.745	22.00	21.860	65.70	174.110	99.30	1386.753	100.00
0.049	0.00	0.389	2.31	3.101	24.00	24.698	69.50	196.714	99.57	1566.785	100.00
0.055	0.00	0.440	2.51	3.503	25.97	27.904	73.25	222.251	99.74	1770.189	100.00
0.062	0.06	0.497	2.79	3.958	27.93	31.527	76.87	251.105	99.86	2000.000	100.00
0.070	0.17	0.561	3.18	4.472	29.87	35.620	80.29	283.704	99.96		

Ti-20Mg_16hrs Yield



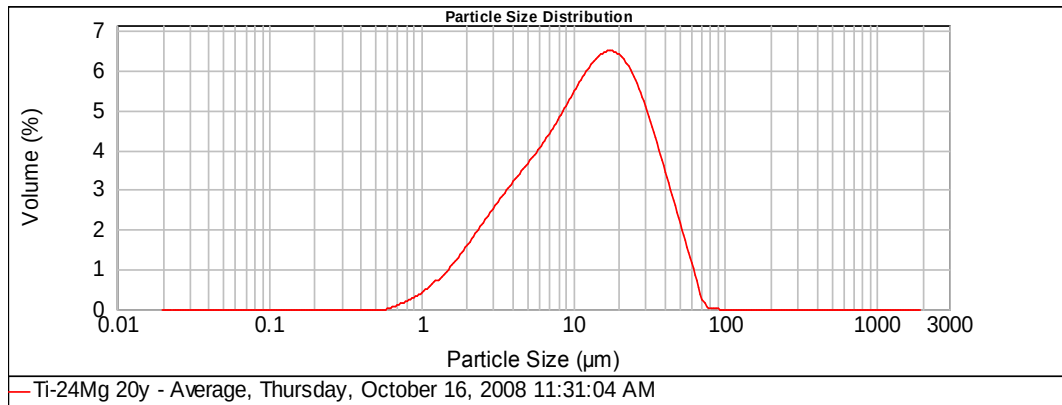
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	0.14	5.053	18.98	40.244	90.69	320.535	100.00
0.011	0.00	0.090	0.00	0.717	0.25	5.709	21.69	45.469	93.15	362.148	100.00
0.013	0.00	0.102	0.00	0.810	0.42	6.450	24.69	51.371	95.11	409.163	100.00
0.014	0.00	0.115	0.00	0.915	0.66	7.287	27.99	58.041	96.61	462.281	100.00
0.016	0.00	0.130	0.00	1.034	1.00	8.233	31.63	65.575	97.71	522.296	100.00
0.018	0.00	0.147	0.00	1.168	1.45	9.302	35.63	74.089	98.50	590.102	100.00
0.021	0.00	0.166	0.00	1.320	2.03	10.510	39.98	83.707	99.05	666.711	100.00
0.024	0.00	0.187	0.00	1.491	2.74	11.874	44.67	94.574	99.41	753.265	100.00
0.027	0.00	0.211	0.00	1.684	3.61	13.416	49.65	106.852	99.65	851.056	100.00
0.030	0.00	0.239	0.00	1.903	4.62	15.157	54.85	120.724	99.80	961.542	100.00
0.034	0.00	0.270	0.00	2.150	5.80	17.125	60.15	136.397	99.86	1086.372	100.00
0.038	0.00	0.305	0.00	2.429	7.15	19.348	65.44	154.104	99.90	1227.408	100.00
0.043	0.00	0.345	0.00	2.745	8.66	21.860	70.61	174.110	99.93	1386.753	100.00
0.049	0.00	0.389	0.00	3.101	10.34	24.698	75.51	196.714	99.96	1566.785	100.00
0.055	0.00	0.440	0.00	3.503	12.21	27.904	80.05	222.251	99.99	1770.189	100.00
0.062	0.00	0.497	0.01	3.958	14.26	31.527	84.12	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	0.06	4.472	16.51	35.620	87.68	283.704	100.00		

Ti-20Mg_20hrs Yield



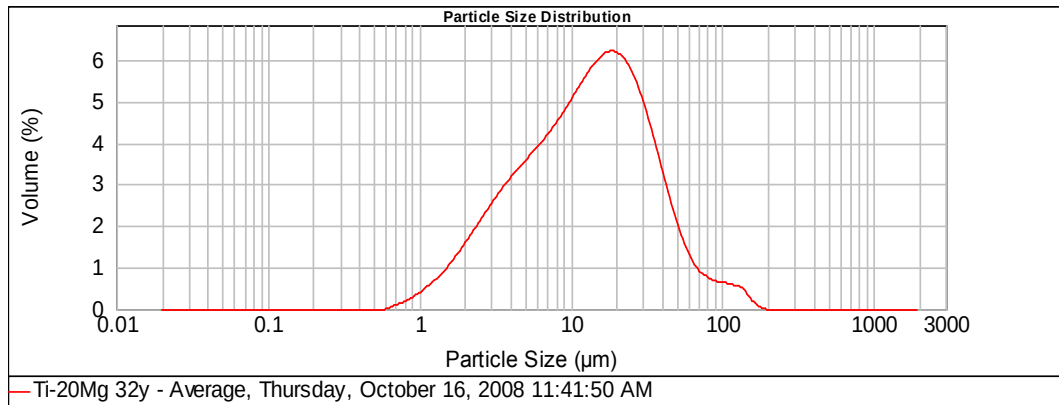
Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	0.03	5.063	22.11	40.244	95.34	320.535	100.00
0.011	0.00	0.090	0.00	0.717	0.12	5.709	25.39	45.469	97.35	362.148	100.00
0.013	0.00	0.102	0.00	0.810	0.25	6.450	28.93	51.371	98.79	409.163	100.00
0.014	0.00	0.115	0.00	0.915	0.47	7.287	32.77	58.041	99.74	462.281	100.00
0.016	0.00	0.130	0.00	1.034	0.79	8.233	36.90	65.575	99.98	522.296	100.00
0.018	0.00	0.147	0.00	1.168	1.23	9.302	41.34	74.089	100.00	590.102	100.00
0.021	0.00	0.166	0.00	1.320	1.81	10.510	46.07	83.707	100.00	666.711	100.00
0.024	0.00	0.187	0.00	1.491	2.56	11.874	51.07	94.574	100.00	753.265	100.00
0.027	0.00	0.211	0.00	1.684	3.50	13.416	56.27	106.852	100.00	851.056	100.00
0.030	0.00	0.239	0.00	1.903	4.63	15.157	61.59	120.724	100.00	961.542	100.00
0.034	0.00	0.270	0.00	2.150	5.99	17.125	66.92	136.397	100.00	1086.372	100.00
0.038	0.00	0.305	0.00	2.429	7.58	19.348	72.15	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	0.00	2.745	9.41	21.860	77.15	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	0.00	3.101	11.47	24.698	81.80	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	0.00	3.503	13.77	27.904	85.99	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	0.00	3.958	16.31	31.527	89.67	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	0.00	4.472	19.09	35.620	92.79	283.704	100.00		

Ti-20Mg_24hrs Yield



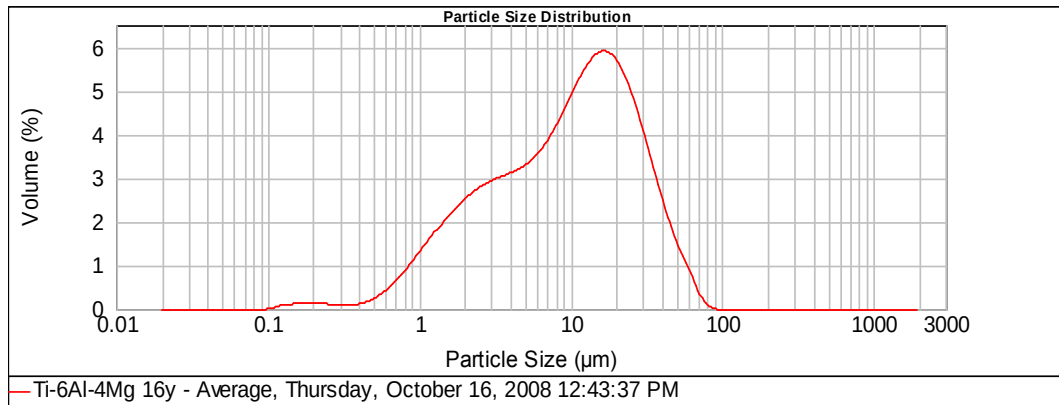
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	0.00	5.053	20.86	40.244	92.75	320.535	100.00
0.011	0.00	0.090	0.00	0.717	0.06	5.709	23.88	45.469	95.31	362.148	100.00
0.013	0.00	0.102	0.00	0.810	0.17	6.450	27.12	51.371	97.32	409.163	100.00
0.014	0.00	0.115	0.00	0.915	0.36	7.287	30.59	58.041	98.76	462.281	100.00
0.016	0.00	0.130	0.00	1.034	0.64	8.233	34.31	65.575	99.68	522.296	100.00
0.018	0.00	0.147	0.00	1.168	1.04	9.302	38.31	74.089	99.98	590.102	100.00
0.021	0.00	0.166	0.00	1.320	1.58	10.510	42.59	83.707	100.00	666.711	100.00
0.024	0.00	0.187	0.00	1.491	2.28	11.874	47.15	94.574	100.00	753.265	100.00
0.027	0.00	0.211	0.00	1.684	3.15	13.416	51.96	106.852	100.00	851.056	100.00
0.030	0.00	0.239	0.00	1.903	4.23	15.157	56.97	120.724	100.00	961.542	100.00
0.034	0.00	0.270	0.00	2.150	5.53	17.125	62.11	136.397	100.00	1086.372	100.00
0.038	0.00	0.305	0.00	2.429	7.06	19.348	67.28	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	0.00	2.745	8.81	21.860	72.37	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	0.00	3.101	10.79	24.698	77.25	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	0.00	3.503	13.00	27.904	81.82	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	0.00	3.958	15.42	31.527	85.97	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	0.00	4.472	18.04	35.620	89.63	283.704	100.00		

Ti-20Mg-32hrs Yield



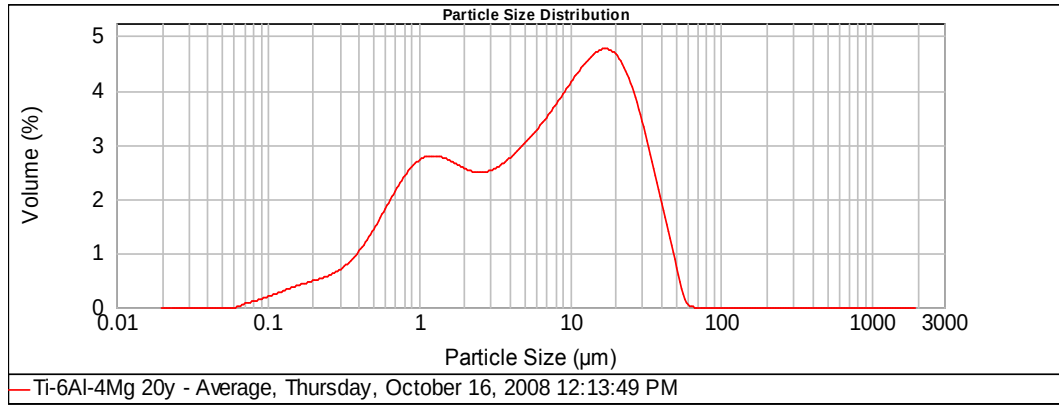
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	0.00	5.053	20.81	40.244	89.48	320.535	100.00
0.011	0.00	0.090	0.00	0.717	0.05	5.709	23.77	45.469	91.91	362.148	100.00
0.013	0.00	0.102	0.00	0.810	0.17	6.450	26.89	51.371	93.78	409.163	100.00
0.014	0.00	0.115	0.00	0.915	0.36	7.287	30.20	58.041	95.19	462.281	100.00
0.016	0.00	0.130	0.00	1.034	0.64	8.233	33.71	65.575	96.22	522.296	100.00
0.018	0.00	0.147	0.00	1.168	1.03	9.302	37.45	74.089	96.99	590.102	100.00
0.021	0.00	0.166	0.00	1.320	1.57	10.510	41.43	83.707	97.61	666.711	100.00
0.024	0.00	0.187	0.00	1.491	2.26	11.874	45.67	94.574	98.15	753.265	100.00
0.027	0.00	0.211	0.00	1.684	3.14	13.416	50.15	106.852	98.66	851.056	100.00
0.030	0.00	0.239	0.00	1.903	4.22	15.157	54.86	120.724	99.14	961.542	100.00
0.034	0.00	0.270	0.00	2.150	5.52	17.125	59.72	136.397	99.58	1086.372	100.00
0.038	0.00	0.305	0.00	2.429	7.05	19.348	64.67	154.104	99.87	1227.408	100.00
0.043	0.00	0.345	0.00	2.745	8.81	21.860	69.59	174.110	99.99	1386.753	100.00
0.049	0.00	0.389	0.00	3.101	10.80	24.698	74.36	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	0.00	3.503	13.01	27.904	78.84	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	0.00	3.958	15.43	31.527	82.91	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	0.00	4.472	18.03	35.620	86.48	283.704	100.00		

Ti-6Al-4Mg_16hrs Yield



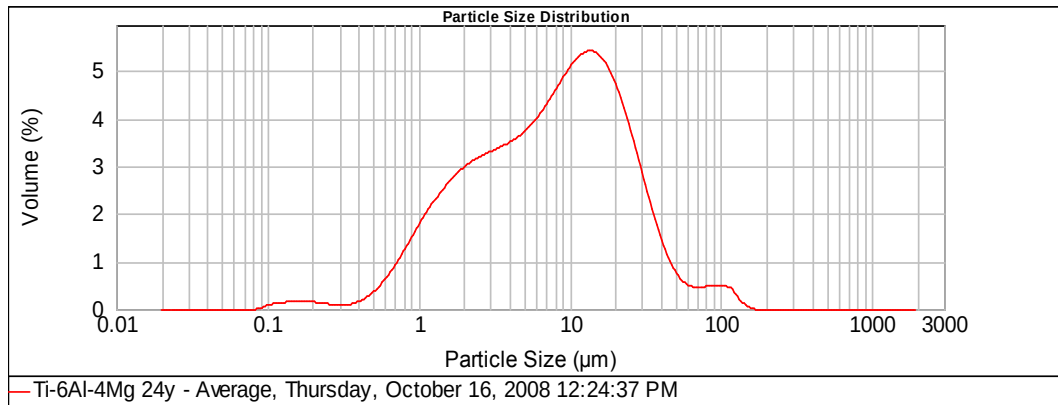
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	1.76	5.053	31.29	40.244	94.63	320.535	100.00
0.011	0.00	0.090	0.00	0.717	2.22	5.709	34.00	45.469	96.46	362.148	100.00
0.013	0.00	0.102	0.00	0.810	2.84	6.450	36.86	51.371	97.84	409.163	100.00
0.014	0.00	0.115	0.02	0.915	3.64	7.287	39.92	58.041	98.85	462.281	100.00
0.016	0.00	0.130	0.09	1.034	4.62	8.233	43.22	65.575	99.58	522.296	100.00
0.018	0.00	0.147	0.18	1.168	5.81	9.302	46.80	74.089	99.90	590.102	100.00
0.021	0.00	0.166	0.29	1.320	7.19	10.510	50.69	83.707	100.00	666.711	100.00
0.024	0.00	0.187	0.40	1.491	8.76	11.874	54.88	94.574	100.00	753.265	100.00
0.027	0.00	0.211	0.52	1.684	10.51	13.416	59.34	106.852	100.00	851.056	100.00
0.030	0.00	0.239	0.62	1.903	12.42	15.157	63.98	120.724	100.00	961.542	100.00
0.034	0.00	0.270	0.72	2.150	14.47	17.125	68.72	136.397	100.00	1086.372	100.00
0.038	0.00	0.305	0.80	2.429	16.64	19.348	73.43	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	0.87	2.745	18.91	21.860	77.98	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	0.95	3.101	21.26	24.698	82.23	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	1.06	3.503	23.67	27.904	86.08	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	1.21	3.958	26.14	31.527	89.46	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	1.43	4.472	28.68	35.620	92.31	283.704	100.00		

Ti-6Al-4Mg_20hrs Yield



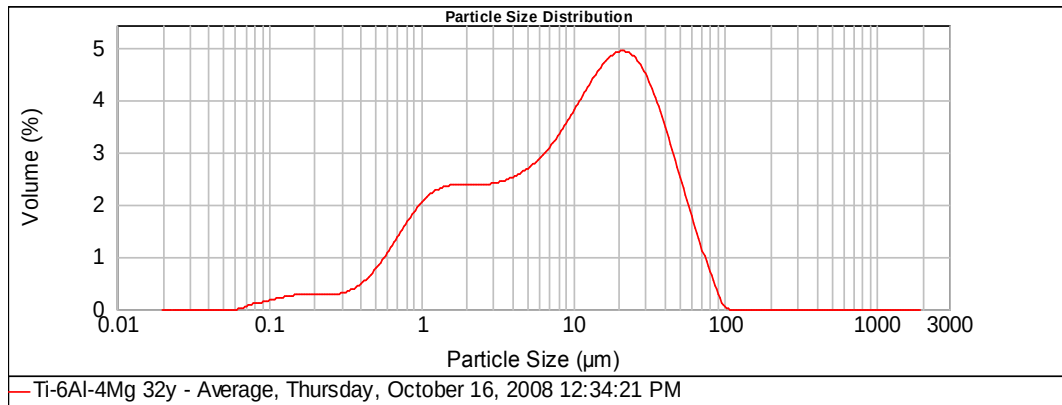
Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.09	0.634	9.22	5.053	44.49	40.244	97.41	320.535	100.00
0.011	0.00	0.090	0.19	0.717	10.85	5.709	46.97	45.469	98.77	362.148	100.00
0.013	0.00	0.102	0.32	0.810	12.67	6.450	49.59	51.371	99.64	409.163	100.00
0.014	0.00	0.115	0.50	0.915	14.66	7.287	52.35	58.041	99.97	462.281	100.00
0.016	0.00	0.130	0.72	1.034	16.79	8.233	55.27	65.575	100.00	522.296	100.00
0.018	0.00	0.147	0.99	1.168	18.99	9.302	58.36	74.089	100.00	590.102	100.00
0.021	0.00	0.166	1.31	1.320	21.22	10.510	61.62	83.707	100.00	666.711	100.00
0.024	0.00	0.187	1.66	1.491	23.43	11.874	65.05	94.574	100.00	753.265	100.00
0.027	0.00	0.211	2.04	1.684	25.59	13.416	68.64	106.852	100.00	851.056	100.00
0.030	0.00	0.239	2.46	1.903	27.68	15.157	72.35	120.724	100.00	961.542	100.00
0.034	0.00	0.270	2.92	2.150	29.72	17.125	76.14	136.397	100.00	1086.372	100.00
0.038	0.00	0.305	3.44	2.429	31.71	19.348	79.93	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	4.03	2.745	33.70	21.860	83.64	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	4.74	3.101	35.70	24.698	87.15	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	5.59	3.503	37.76	27.904	90.37	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	6.60	3.958	39.90	31.527	93.19	251.105	100.00	2000.000	100.00
0.070	0.02	0.561	7.80	4.472	42.14	35.620	95.55	283.704	100.00		

Ti-6Al-4Mg_24hrs Yield



Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	0.080	0.00	0.634	2.35	5.053	37.31	40.244	95.15	320.535	100.00
0.011	0.00	0.090	0.00	0.717	3.01	5.709	40.36	45.469	96.18	362.148	100.00
0.013	0.00	0.102	0.05	0.810	3.88	6.450	43.56	51.371	96.90	409.163	100.00
0.014	0.00	0.115	0.14	0.915	4.98	7.287	46.95	58.041	97.41	462.281	100.00
0.016	0.00	0.130	0.24	1.034	6.30	8.233	50.56	65.575	97.81	522.296	100.00
0.018	0.00	0.147	0.36	1.168	7.86	9.302	54.38	74.089	98.18	590.102	100.00
0.021	0.00	0.166	0.50	1.320	9.64	10.510	58.41	83.707	98.55	666.711	100.00
0.024	0.00	0.187	0.63	1.491	11.61	11.874	62.61	94.574	98.96	753.265	100.00
0.027	0.00	0.211	0.76	1.684	13.74	13.416	66.91	106.852	99.35	851.056	100.00
0.030	0.00	0.239	0.86	1.903	16.03	15.157	71.23	120.724	99.72	961.542	100.00
0.034	0.00	0.270	0.95	2.150	18.42	17.125	75.46	136.397	99.92	1086.372	100.00
0.038	0.00	0.305	1.02	2.429	20.92	19.348	79.50	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	1.09	2.745	23.48	21.860	83.23	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	1.18	3.101	26.11	24.698	86.57	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	1.32	3.503	28.80	27.904	89.45	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	1.54	3.958	31.56	31.527	91.84	251.105	100.00	2000.000	100.00
0.070	0.00	0.561	1.87	4.472	34.38	35.620	93.72	283.704	100.00		

Ti-6Al-4Mg_32hrs Yield



Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	0.080	0.09	0.634	5.19	5.053	35.25	40.244	90.07	320.535	100.00
0.011	0.00	0.090	0.18	0.717	6.22	5.709	37.45	45.469	92.72	362.148	100.00
0.013	0.00	0.102	0.30	0.810	7.43	6.450	39.76	51.371	94.94	409.163	100.00
0.014	0.00	0.115	0.45	0.915	8.83	7.287	42.20	58.041	96.75	462.281	100.00
0.016	0.00	0.130	0.63	1.034	10.40	8.233	44.79	65.575	98.15	522.296	100.00
0.018	0.00	0.147	0.84	1.168	12.10	9.302	47.57	74.089	99.14	590.102	100.00
0.021	0.00	0.166	1.06	1.320	13.90	10.510	50.55	83.707	99.74	666.711	100.00
0.024	0.00	0.187	1.29	1.491	15.76	11.874	53.74	94.574	99.99	753.265	100.00
0.027	0.00	0.211	1.52	1.684	17.66	13.416	57.14	106.852	100.00	851.056	100.00
0.030	0.00	0.239	1.74	1.903	19.56	15.157	60.73	120.724	100.00	961.542	100.00
0.034	0.00	0.270	1.97	2.150	21.46	17.125	64.49	136.397	100.00	1086.372	100.00
0.038	0.00	0.305	2.20	2.429	23.36	19.348	68.36	154.104	100.00	1227.408	100.00
0.043	0.00	0.345	2.46	2.745	25.26	21.860	72.30	174.110	100.00	1386.753	100.00
0.049	0.00	0.389	2.78	3.101	27.18	24.698	76.22	196.714	100.00	1566.785	100.00
0.055	0.00	0.440	3.18	3.503	29.12	27.904	80.04	222.251	100.00	1770.189	100.00
0.062	0.00	0.497	3.70	3.958	31.11	31.527	83.68	251.105	100.00	2000.000	100.00
0.070	0.01	0.561	4.36	4.472	33.15	35.620	87.05	283.704	100.00		

Appendix B: Tables of green densities

Ti-10Mg alloy powders

Milling time (hrs)	Green Density (27MPa), g/cm³	Green Density (33MPa), g/cm³
16	2.75	2.83
20	2.65	2.71
24	2.70	2.79
32	2.70	2.75

Ti-20Mg alloy powders

Milling time (hrs)	Green Density (27MPa), g/cm³	Green Density (33MPa), g/cm³
16	2.59	2.72
20	2.54	2.67
24	2.58	2.72
32	2.58	2.69

Ti-6Al-4Mg alloy powders

Milling time (hrs)	Green Density (27MPa), g/cm³	Green Density (33MPa), g/cm³
16	2.66	2.74
20	2.62	2.70
24	2.68	2.73
32	2.64	2.72

Appendix C: Calculating theoretical density using the Rule of Mixtures.

$$\rho_{th} = W_{tot} / [(w_a/r_a) + (w_b/r_b) + \dots] \text{-----Equation E1}$$

Where W_{tot} is the total weight of mixture (g),

$w_a, w_b, \dots$ are the weights of components in the mixture (g),

$r_a, r_b, \dots$ are the densities of components in the mixture (g/cm^3).

Basis in these calculations: 100g of mixture; i.e. $W_{tot}=100\text{g}$.

$$r_{Ti}=4.51\text{g/cm}^3, r_{Mg}=1.74\text{g/cm}^3, r_{Al}=2.70\text{g/cm}^3$$

(a) Theoretical density of Ti-10Mg

$$\rho_{th} = 100 / [(w_{Ti}/r_{Ti}) + (w_{Mg}/r_{Mg})] \text{ g/cm}^3$$

$$\rho_{th} = 100 / [(90/4.51) + (10/1.74)] \text{ g/cm}^3 = 3.89 \text{ g/cm}^3.$$

$$\text{Theoretical density of Ti-10Mg} = 3.89 \text{ g/cm}^3.$$

(b) Theoretical density of Ti-20Mg

$$\rho_{th} = 100 / [(w_{Ti}/r_{Ti}) + (w_{Mg}/r_{Mg})] \text{ g/cm}^3$$

$$\rho_{th} = 100 / [(80/4.51) + (20/1.74)] \text{ g/cm}^3 = 3.42 \text{ g/cm}^3.$$

$$\text{Theoretical density of Ti-20Mg} = 3.42 \text{ g/cm}^3.$$

(c) Theoretical density of Ti-6Al-4Mg

$$\rho_{th} = 100 / [(w_{Ti}/r_{Ti}) + (w_{Mg}/r_{Mg}) + (w_{Al}/r_{Al})] \text{ g/cm}^3$$

$$\rho_{th} = 100 / [(90/4.51) + (6/2.70) + (4/1.74)] \text{ g/cm}^3 = 4.08 \text{ g/cm}^3.$$

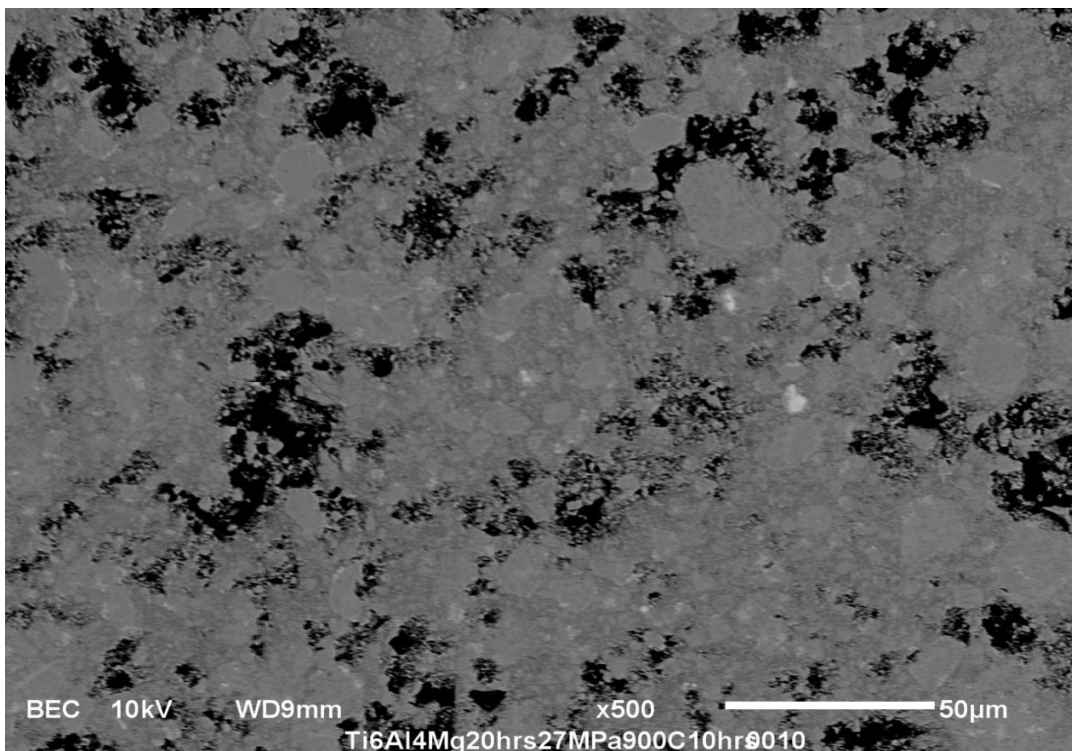
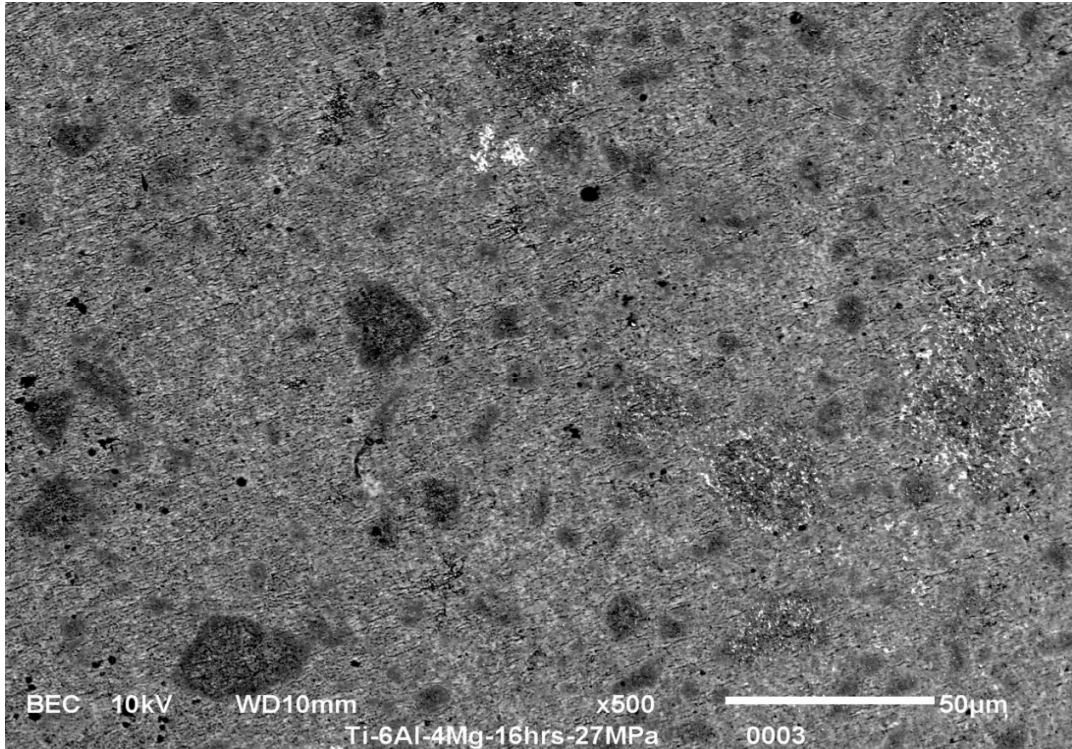
$$\text{Theoretical density of Ti-6Al-4Mg} = 4.08 \text{ g/cm}^3.$$

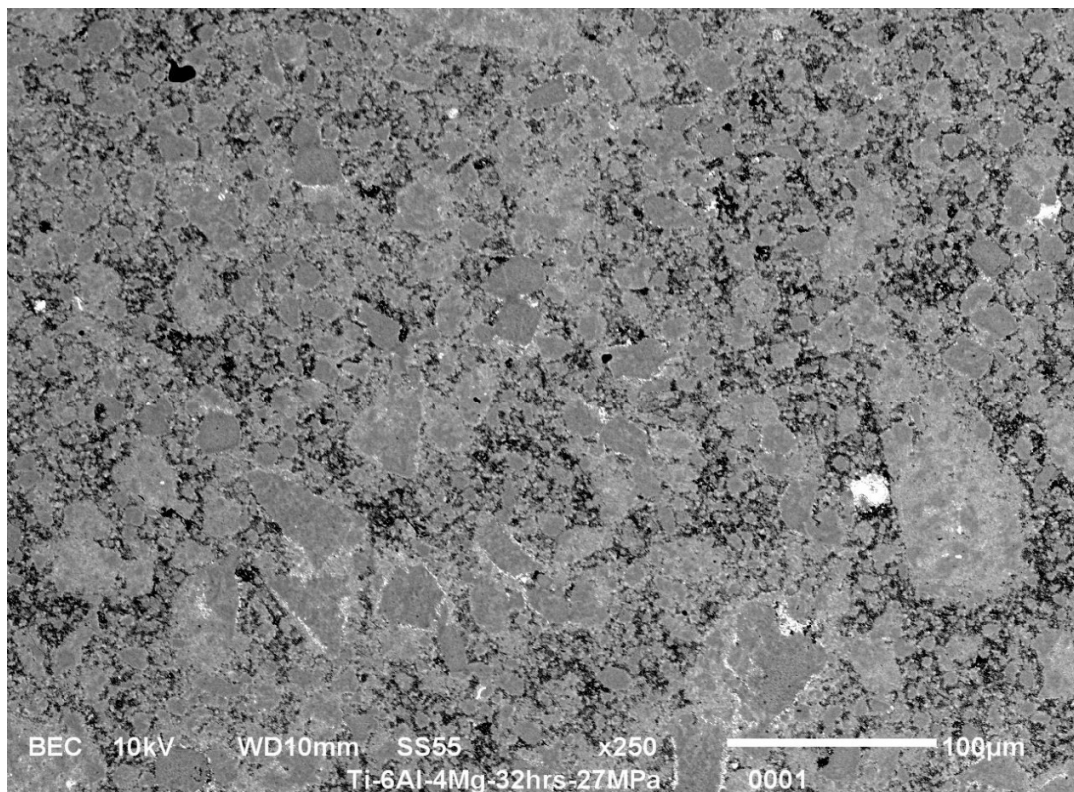
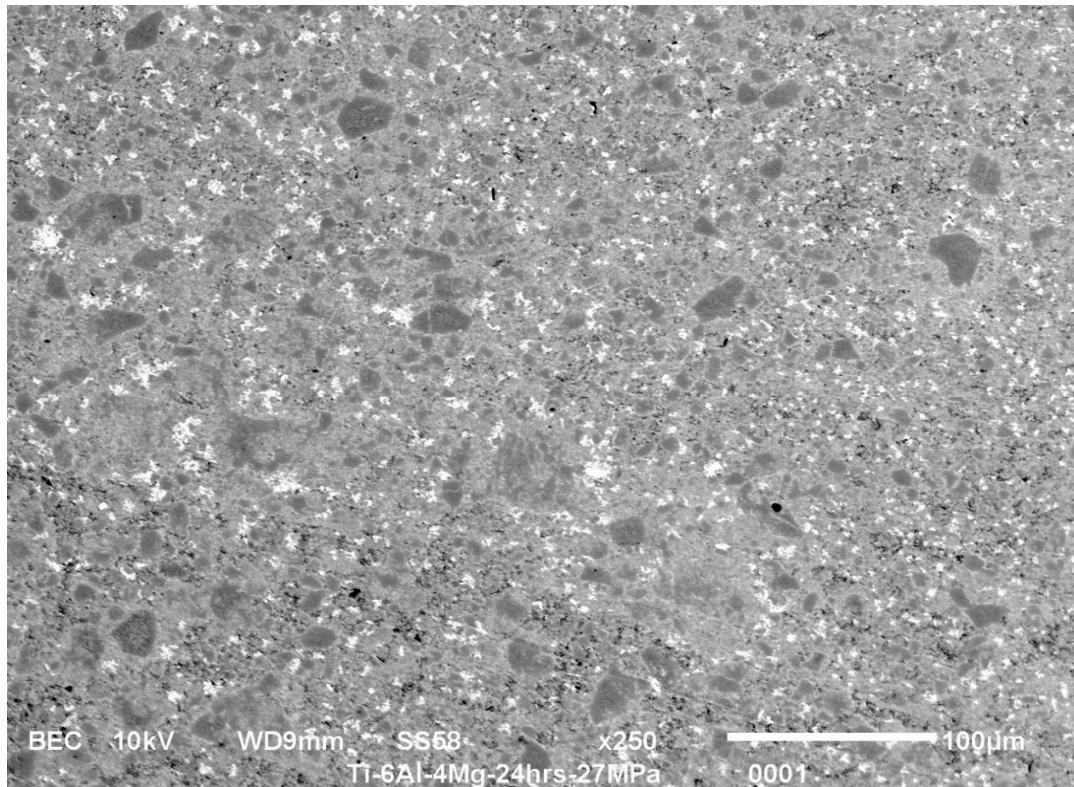
Percent theoretical densities were calculated as follows:

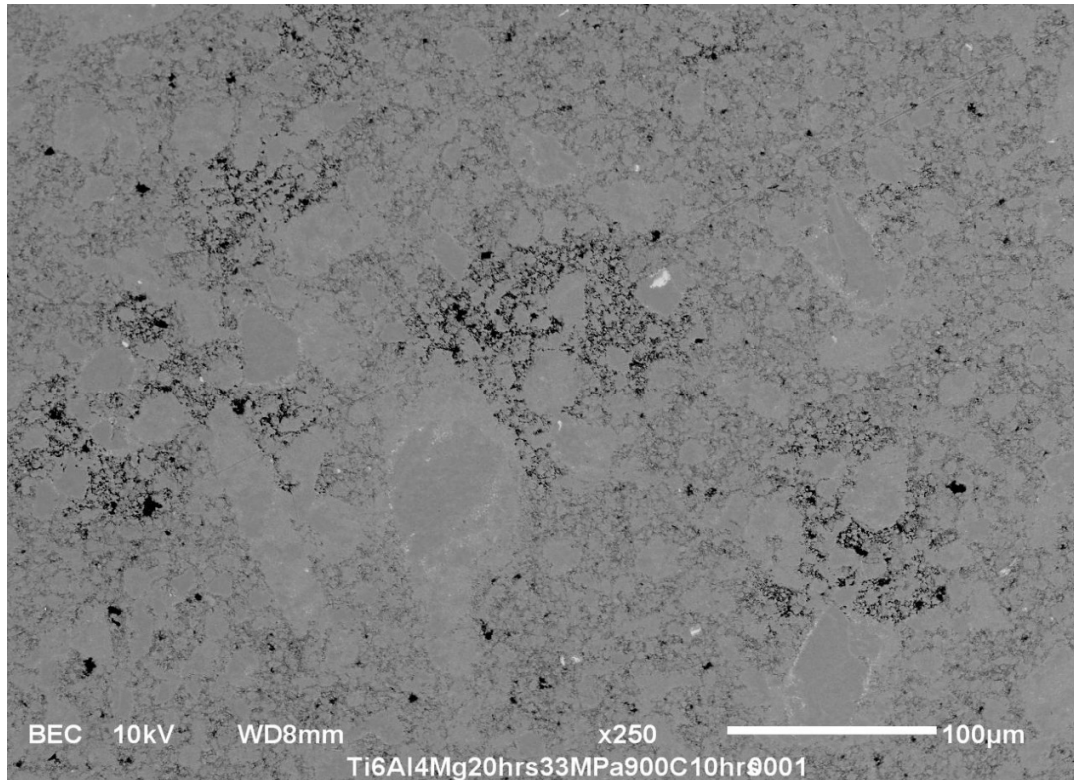
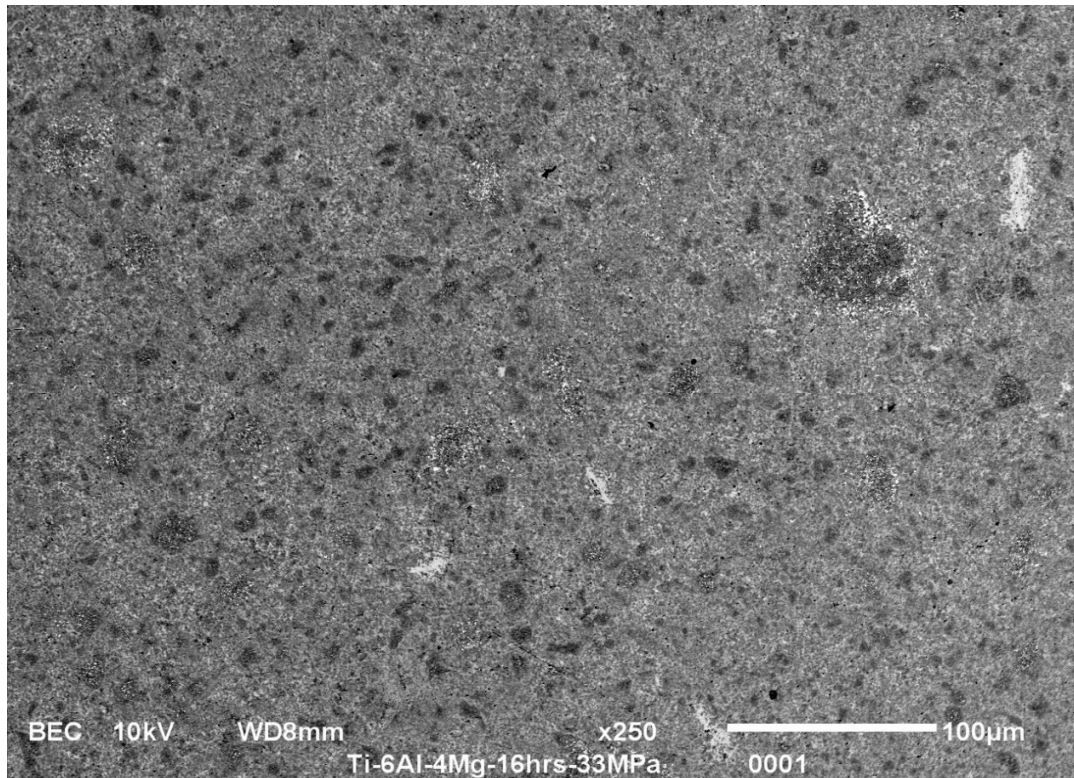
% theoretical density= (green density/theoretical density) x100

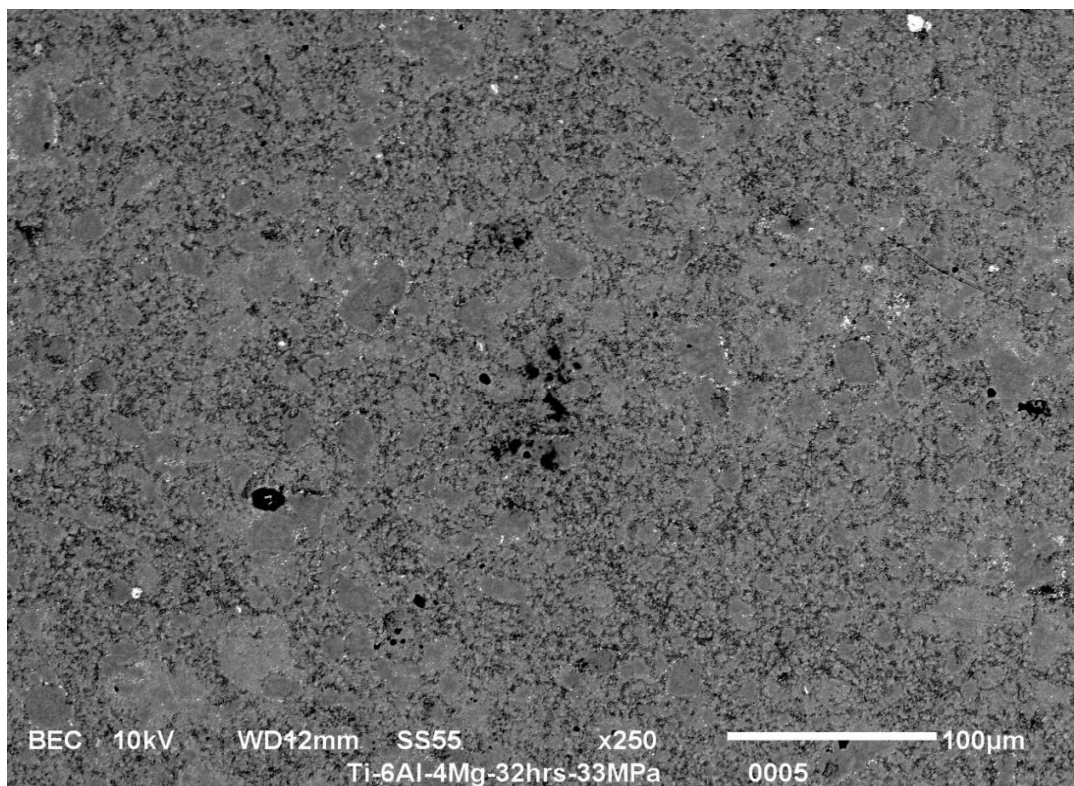
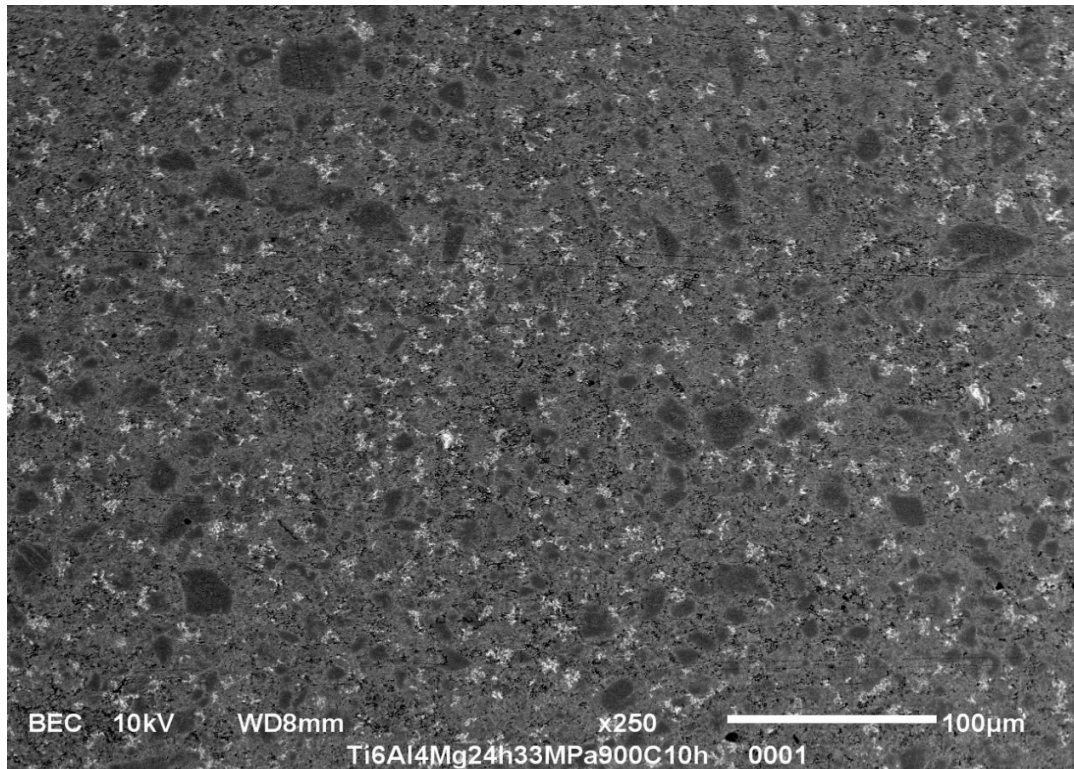
Alloy	% Theoretical density (27MPa)	% Theoretical density (33MPa)
Ti-10Mg 16h	70.7	72.8
Ti-10Mg 20h	68.1	69.7
Ti-10Mg 24h	69.4	71.7
Ti-10Mg 32h	69.4	70.7
Ti-20Mg 16h	75.7	79.5
Ti-20Mg 20h	74.3	78.1
Ti-20Mg 24h	75.4	79.5
Ti-20Mg 32h	75.4	78.7
Ti-6Al-4Mg 16h	65.2	67.2
Ti-6Al-4Mg 20h	64.2	66.2
Ti-6Al-4Mg 24h	65.7	66.9
Ti-6Al-4Mg 32h	64.7	66.7

Appendix D: SEM Images for sintered Ti-6Al-4Mg specimens









Appendix E: Tables of hardness values

Ti-10Mg specimens

Alloy	Hardness (HV)										Average (HV)
Ti-10Mg_16h_27MPa	246.6	285.5	256.3	254.8	260.4	236.9	240.5	239.6	229.2	233.7	248.4
Ti-10Mg_20h_27MPa	159.8	209.1	163.1	189.8	170.9	150.3	141.9	132.5	128.5	133.6	156.0
Ti-10Mg_24h_27MPa	387.9	373.8	346.4	386.2	362.2	353.5	344.1	359.3	397.6	351.6	366.3
Ti-10Mg_32h_27MPa	416.0	413.7	379.5	353.5	341.6	378.8	400.5	407.7	386.7	384.7	386.3
Ti-10Mg_16h_33MPa	392.2	375.7	375.7	377.4	367.9	376.2	362.2	405.3	368.4	384.9	378.6
Ti-10Mg_20h_33MPa	182.3	169.6	172.3	181.5	200.7	162.8	190.8	175.4	167.0	157.9	176.0
Ti-10Mg_24h_33MPa	404.5	396.6	398.4	414.3	389.7	399.0	371.4	392.0	397.1	389.9	395.3
Ti-10Mg_32h_33MPa	442.8	413.2	400.0	436.5	425.6	389.4	439.8	440.4	448.4	407.2	424.3

Ti-20Mg specimens

Alloy	Hardness (HV)										Average (HV)
Ti-20Mg_16h_27MPa	272.5	262.3	261.4	264.9	262.6	283.6	279.8	285.9	276.3	258.7	270.8
Ti-20Mg_20h_27MPa	264.3	277.8	268.4	295.6	258.7	247.0	297.0	282.2	277.2	275.7	274.4
Ti-20Mg_24h_27MPa	294.1	286.5	259.3	293.1	331.1	277.5	304.3	299.8	310.0	308	296.4
Ti-20Mg_32h_27MPa	367.0	343.3	340.4	349.2	342.9	316.9	337.1	333.9	360.2	351.6	344.3
Ti-20Mg_16h_33MPa	293.0	311.2	325.2	316.0	312.3	311.8	319.5	297.3	300.2	317.3	310.4
Ti-20Mg_20h_33MPa	322.5	291.8	321.2	322.3	299.7	332.7	310.9	288.3	285.2	299.7	307.4
Ti-20Mg_24h_33MPa	378.8	337.1	350.9	335.7	325.6	346.4	337.1	335.3	347.5	339.2	343.4
Ti-20Mg_32h_33MPa	450.6	432.3	423.3	433.8	408.5	408.8	429.4	425.6	431.4	430.5	427.4

Ti-6Al-4Mg specimens

Alloy	Hardness (HV)										Average (HV)
Ti-6Al-4Mg_16h_27MPa	859.1	902.9	978.8	872.1	1012.0	984.1	916.6	847	907.8	859.9	914.0
Ti-6Al-4Mg_20h_27MPa	325.8	324.6	351.3	341.0	397.1	290.4	372.1	370.5	295.6	382.2	345.0
Ti-6Al-4Mg_24h_27MPa	602.0	823.6	631.2	579.0	597.2	745.8	617.9	649.3	679.1	726.1	665.1
Ti-6Al-4Mg_32h_27MPa	686.1	695.0	664.2	623.0	695.0	769.8	658.6	615.9	687.3	665.9	676.1
Ti-6Al-4Mg_16h_33MPa	953.0	980.4	979.4	953.7	1002.9	884.0	839.1	841.2	817.8	873.8	912.5
Ti-6Al-4Mg_20h_33MPa	584.1	581.8	602.1	574.1	586.0	603.6	611.2	580.4	664.8	630.2	601.8
Ti-6Al-4Mg_24h_33MPa	724.9	754.9	715.6	659.1	687.2	733.8	774.5	873.6	824.7	801.5	755.0
Ti-6Al-4Mg_32h_33MPa	775.3	773.7	784.9	758.4	810.6	784.7	807	836.2	905	833.8	807.0

Appendix F: Tables of sintered densities**Determination of sintered densities: Archimedes Principle**

$$\rho_s = (m_s \cdot \rho_w) / (m_s - m_w)$$

Where ρ_s = sintered density (g/cm^3), ρ_w = density of water at room temperature ($\sim 1 \text{ g/cm}^3$), m_s = mass of the sintered body in air (g) and m_w = mass of sintered body in water (g).

Thus, the formula simplifies to $\rho_s = (m_s) / (m_s - m_w)$ since $\rho_w = 1 \text{ g/cm}^3$

Specimen	Dry mass(g)	Suspended weight (g)	Sintered Density (g/cm^3)
Ti-10Mg 16h_27MPa	1.814	1.422	4.62
Ti-10Mg 20h_27MPa	1.772	1.314	3.89
Ti-10Mg 24h_27MPa	1.824	1.408	4.38
Ti-10Mg 32h_27MPa	1.821	1.410	4.43
Ti-10Mg 16h_33MPa	1.831	1.398	4.23
Ti-10Mg 20h_33MPa	1.841	1.412	4.29
Ti-10Mg 24h_33MPa	1.848	1.414	4.26
Ti-10Mg 32h_33MPa	1.824	1.406	4.36
Ti-20Mg 16h_27MPa	1.654	1.284	4.47
Ti-20Mg 20h_27MPa	1.634	1.260	4.37
Ti-20Mg 24h_27MPa	1.625	1.251	4.34
Ti-20Mg 32h_27MPa	1.657	1.270	4.28
Ti-20Mg 16h_33MPa	1.686	1.286	4.21
Ti-20Mg 20h_33MPa	1.648	1.285	4.54

Ti-20Mg 24h_33MPa	1.662	1.279	4.34
Ti-20Mg 32h_33MPa	1.675	1.283	4.27
Ti-6Al-4Mg 16h_27MPa	1.861	1.436	4.38
Ti-6Al-4Mg 20h_27MPa	1.866	1.436	4.34
Ti-6Al-4Mg 24h_27MPa	1.882	1.455	4.41
Ti-6Al-4Mg 32h_27MPa	1.882	1.450	4.36
Ti-6Al-4Mg 16h_33MPa	1.905	1.469	4.37
Ti-6Al-4Mg 20h_33MPa	1.898	1.477	4.51
Ti-6Al-4Mg 24h_33MPa	1.886	1.461	4.43
Ti-6Al-4Mg 32h_33MPa	1.904	1.465	4.33

Ti-10Mg specimens

Milling time (hrs)	Sintered Density (27MPa), g/cm³	Sintered Density (33MPa), g/cm³
16	4.62	4.23
20	3.89	4.29
24	4.38	4.26
32	4.43	4.36

Ti-20Mg specimens

Milling time (hrs)	Sintered Density (27MPa), g/cm³	Sintered Density (33MPa), g/cm³
16	4.47	4.21
20	4.37	4.54
24	4.34	4.34
32	4.28	4.27

Ti-6Al-4Mg specimens

Milling time (hrs)	Sintered Density (27MPa), g/cm³	Sintered Density (33MPa), g/cm³
16	4.38	4.37
20	4.34	4.51
24	4.41	4.43
32	4.36	4.33