various precipitates.

3.6.3 Transmission electron microscopy

Thin foils were examined in a Jeol 100C and 200C transmission electron microscopes.

Thin foils were prepared from discs cut with a diamond saw from 3,1mm rod machined from broken Charpy specimens. The discs which were initially 200µm thick, were ground down to a thickness of 80µm on 1200 grade emery paper. The final thinning was carried out in a Struers Tenupol twin jet polisher. The electropolishing solution used was:

6% perchloric acid:
94% glacial acetic acid: and
a small amount of iron slag to facilitate the reduction of chromium.

The polishing conditions were, a flow rate setting of 0.5, a voltage of 45V and a current of 100mA. Polishing was carried out at ambient temperature.

Three specimens were selected for examination by transmission electron microscopy. These were two 0,1% REM- containing alloys, one ductile and one brittle, and one base alloy (no REM) which was ductile.

3.6.4 Microprobe

Four specimens: V1, V2/1, V4/0 (all containing no REMs) and V2/3 (0,6%REM) were examined in the GEOL 733 microprobe. A qualitative analysis of the precipitates in the alloys was carried out. The relative concentrations of the elements such as oxygen were also estimated by comparing the magnitude of the deflection on the indicator guage corresponding to the different elements. For example, a large deflection indicated a higher concentration than a smaller deflection.

The specimens studied were mounted in copper and examined in the as-polished (1μm finish) but unetched condition.

3.7 IMAGE ANALYSIS

The IBAS interactive image analysis system was used to investigate the differences in inclusion distribution, size and morphology in six samples.

The precipitates in each sample were divided into two classes: needle-like and round. The number, volume fraction, size distribution and roundness of the precipitates in each class were estimated.

The computer programme used for the analysis is given in the Appendix C.

4 RESULTS AND PRELIMINARY DISCUSSION

4.1 RARE EARTH METAL YIELDS

Two methods of analysis (graviometric or atomic bombardment) were used to estimate REM yields. The results ootained are reproduced in Table 4.1.

Table 4.1 Rare Earth addition and recovery

Alloy	REM added	REM recovered Graviometry	Atomic bombardment
V2/3	0,15	-	0
V2/2/2	0,3	-	0
V2/3	0,6	-	0,32
V2/4	0,9	-	0,6
V4/0,05	0,05	0,04	-
V4/0,10	0,10	0,06	-
V4/0,15	0,15	0,04	-

\- method not used

Both sets of results were considered dubious. The graviometric analyses which produce very similar values, reflect very large differences in the recovery rate of REMs: and atomic bombardment analyses indicate that alloys V2/3 and V2/2/2 have zero REM content despite the presence of numerous REM inclusions (which were identified using the microprobe).

Furthermore, a correlation between methods of rare earth addition, recovery and analysis has not yet been documented[72], and it has also been reported by Gulyaev et al[76] and Luyckx et al[77] that rare earth recovery may vary between 5 and 95% depending on the method or type of addition. As a consequence, the amount of REM added to a melt rather than REM yield is quoted in reference to any alloy studied in the current programme.

4.2 CHARPY TESTS

Charpy impact tests have been used routinely in evaluating the toughness of ferritic stainless steels. Previous studies have shown that the DBTT increases with increasing amounts of interstitials in solution for medium and high interstitial levels. In the current study impact tests were carried out to investigate this effect in the present steels and to allow conparison of the results obtained with those of previous studies. It is also of interest to compare the DBTT results of the Charpy and slow bend testing methods.

4.2.1 Fabrication route alloys

Alloy V1 was subjected to Charpy impact tests after different annealing treatments. The annealing treatment and results of the tests are given in Table 4.2.

Table 4.2 Alloy V1 - Charpy impact tests results

Annealing temperature(°C)	Time (minutes)	Impact Energy (Joules)
950	30	42
950	60	47,5
1100	30	23
1100	60	23

It is evident from this table that the alloy annealed at 950 °C for 60 minutes gave the highest impact energy. This treatment was then used as the basis for the heat treatment of further alloys.

4.2.2 Alloys V2 and V3

The DBTT curves of the V2 and V3 alloys containing 0, 0,15 and 0,3 percent REM are shown in Figures 4.1 to 4.3. Although an upper and lower shelf level are clearly distinguishable, there is a wide scatter in the impact values. This effect is discussed in the next section. Table 4.3 summarises the inferences drawn from the results.

Table 4.3 Alloys V2 and V3 - Charpy test results

Alloys	%REM added	DBTT	Transition temperature range, °C
V2/1/2	0	-10	20
V3/2	0,15	-12	10
V2/2/2	0,3	15	30

The results indicate that an optimum REM level exists. This effect is illustrated in Figure 4.4. For a REM addition of 0,15% a slight improvement in the DBTT is obtained, and the transition range is considerably narrowed. It should however be noted that the total interstitial contents (C, O and N) for the alloys V2/1/2, V3/2 and V2/2/2 are 0,046: 0,038 and 0,045 % respectively. The alloy containing 0,15% REM therefore also has the lowest interstitial level and the difference (0,007%) corresponds to a reduction in the oxygen content. Thus, the reduction in the DBTT may not necessarily be directly attributable to the REM content.

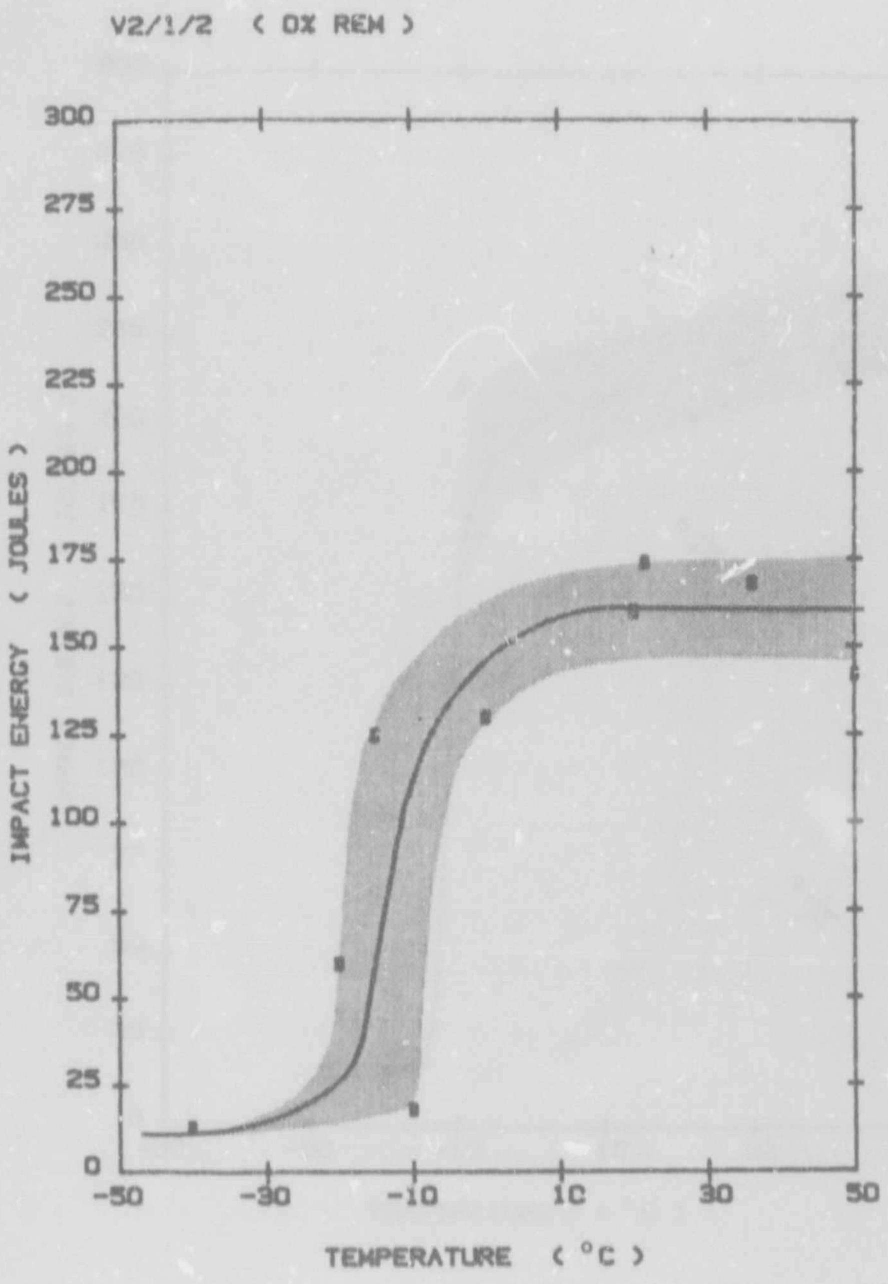

Figure 4.1 Charpy test results: DBTT curve of the V2/1/2 alloy.

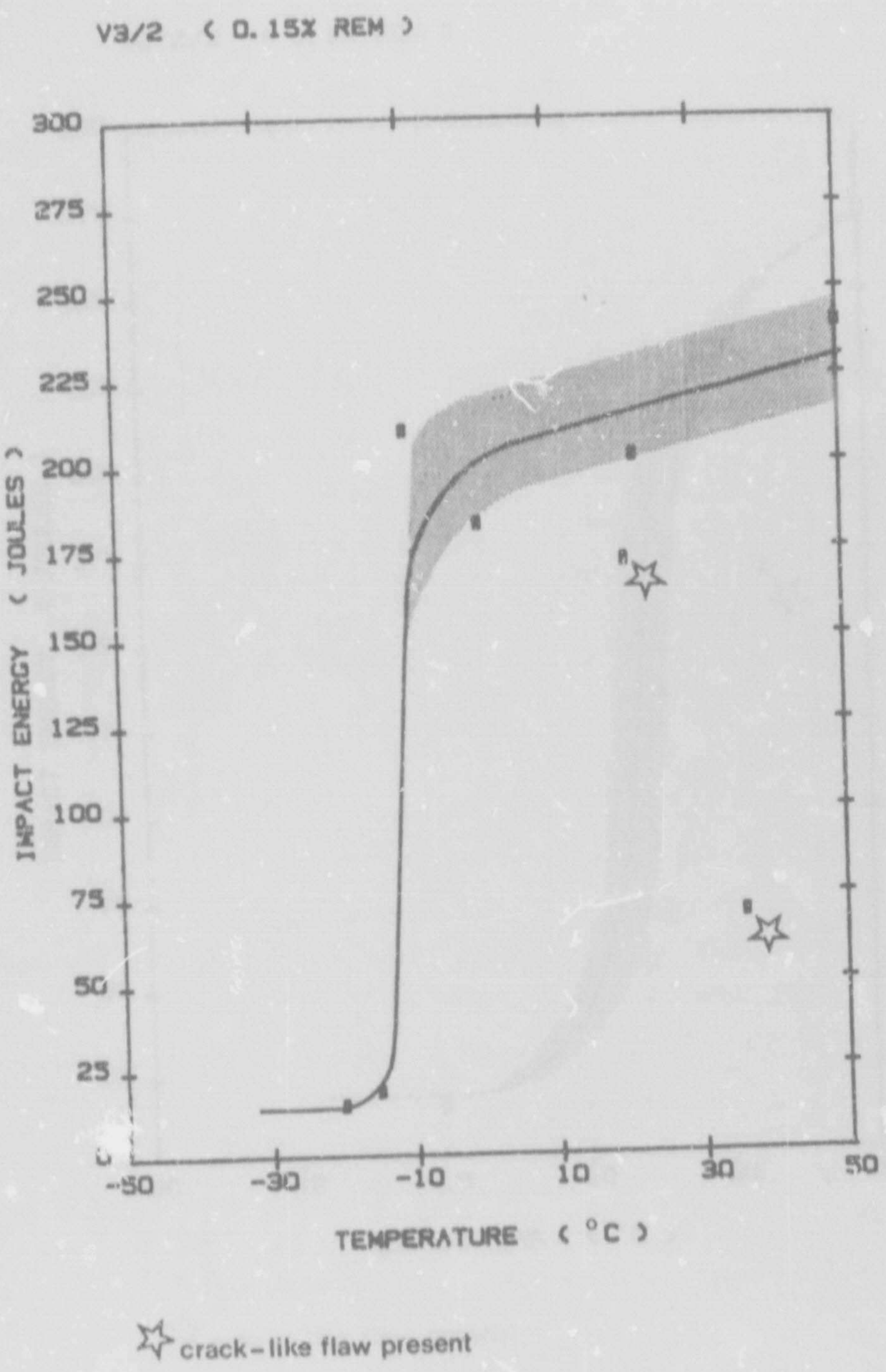

Figure 4.2 Charpy test results: DBTT curve of the V3/2 alloy.

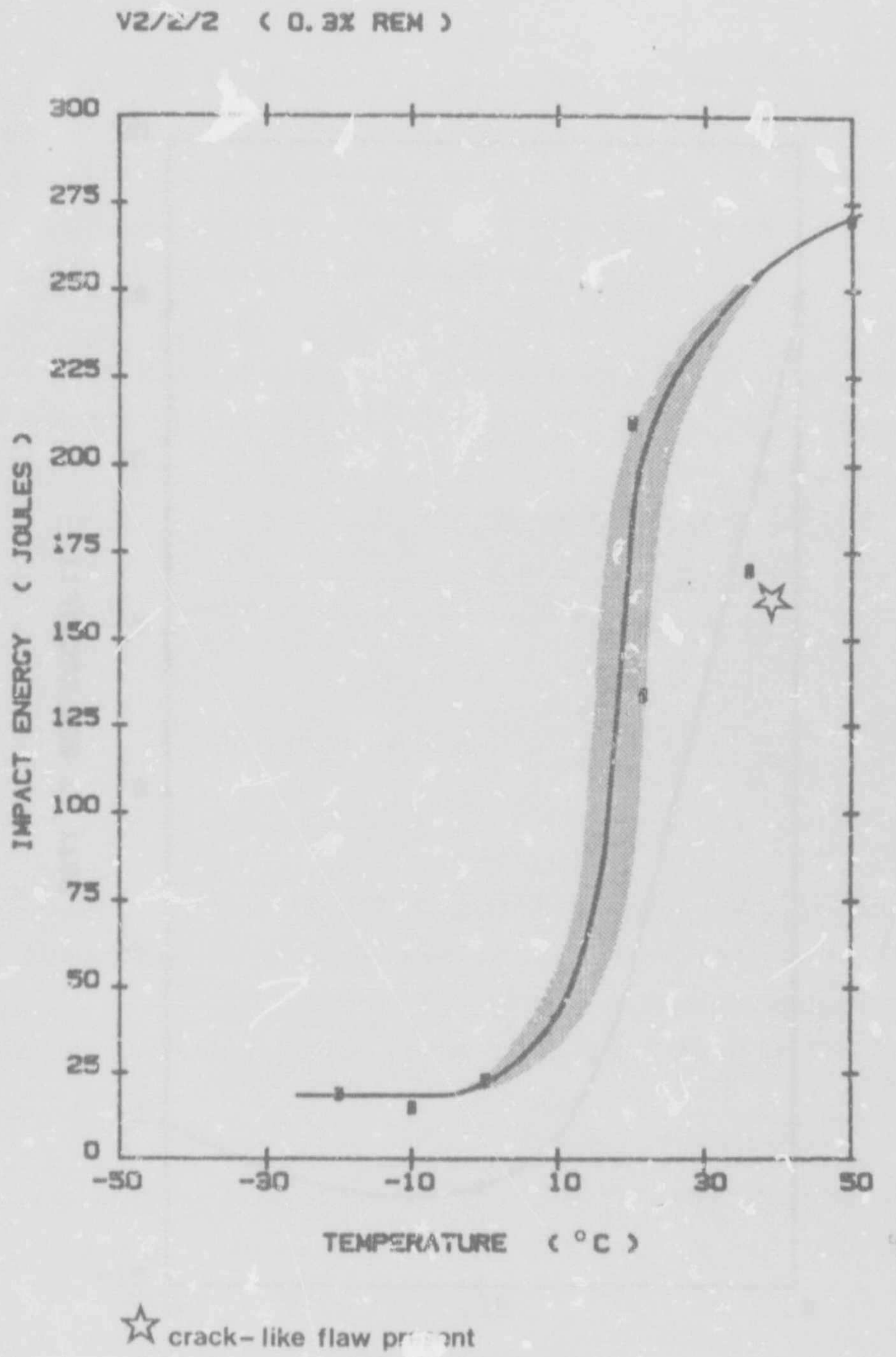

Figure 4.3 Charpy test results: DBTT curve of the V2/2/2 alloy.

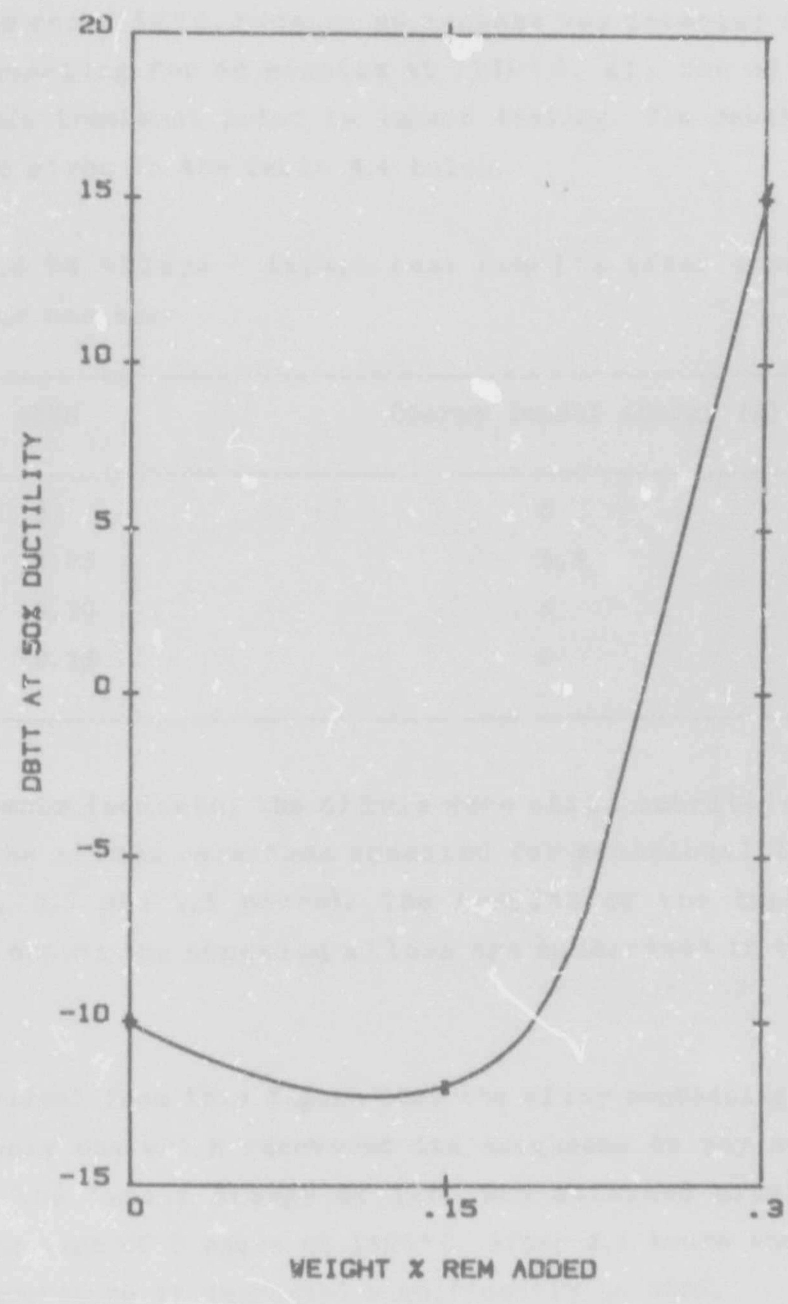

Figure 4.4 Charpy test results: DBTT versus content.

4.2.3 The V4 alloys

After rolling the V4 alloys were subjected to annealing trials at 950, 1050 and 1150 °C. Because no σ-phase was detected optically after annealing for 60 minutes at 1150 °C, all the alloys were given this treatment prior to impact testing. The results of the tests are given in the Table 4.4 below.

Table 4.4 V4 alloys - impact test results after annealing at 1150°C for one hour

%REM	Charpy impact energy (J)
0	6
0,05	5,5
0,10	6
0,15	6

As the tests indicate, the alloys were still embrittled at this stage. The alloys were then annealed for additional lengths of time (1, 1.5 and 2.5 hours). The results of the impact tests carried out on the annealed alloys are summarised in the Figure 4.5.

It is evident from this figure that the alloy containing 0,10% REM is the only one which recovered its toughness to any significant extent. The impact energy of 140J was attained after a total annealing time of 2 hours at 1150 °C. After 3,5 hours annealing at this temperature it decreased significantly to 100J.

The DBTT of the V4/0,10 alloy was determined for the 2 hour anneal at 1150 °C (Figure 4.6).

The results show a large scatter for the impact tests carried at ambient temperature. The DBTT for the alloy (0 °C) is slightly higher than that for the V2 and V3 alloys containing 0 and 0,15%

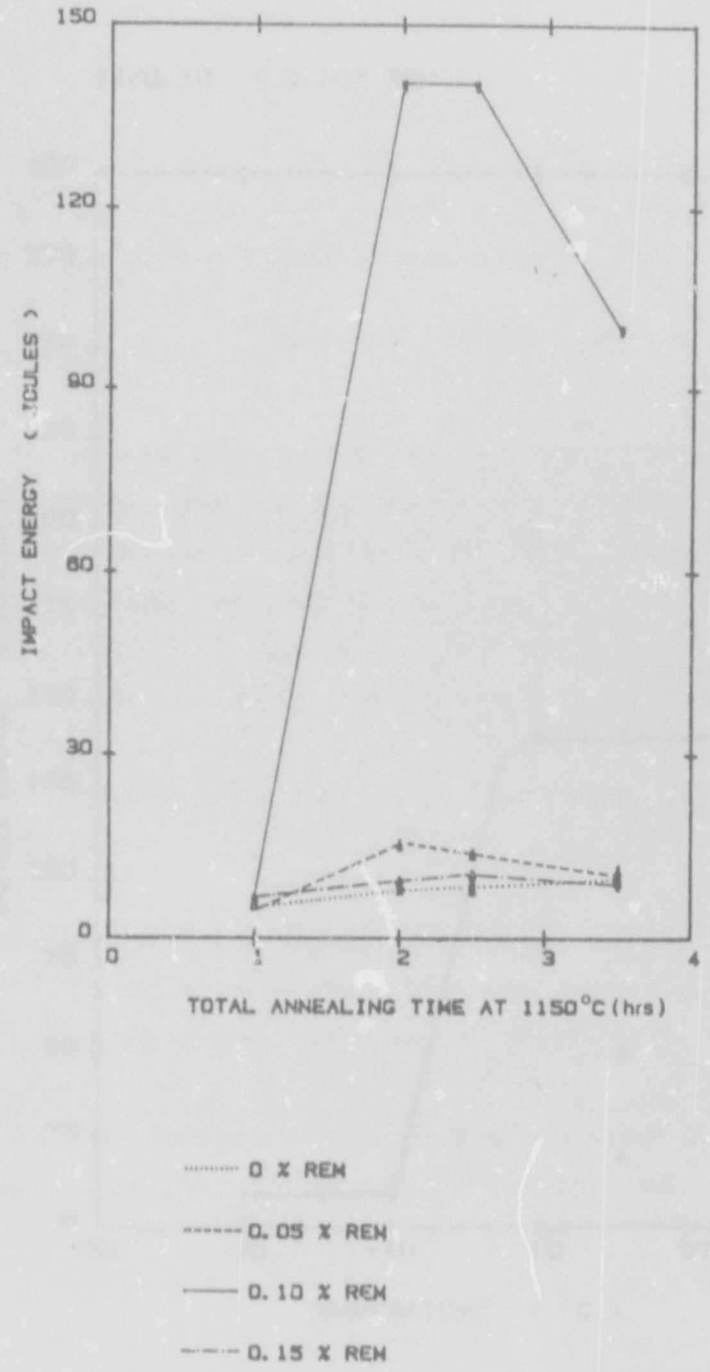

Figure 4.5 V4 alloys. Impact energies after annealing at 1150°C.

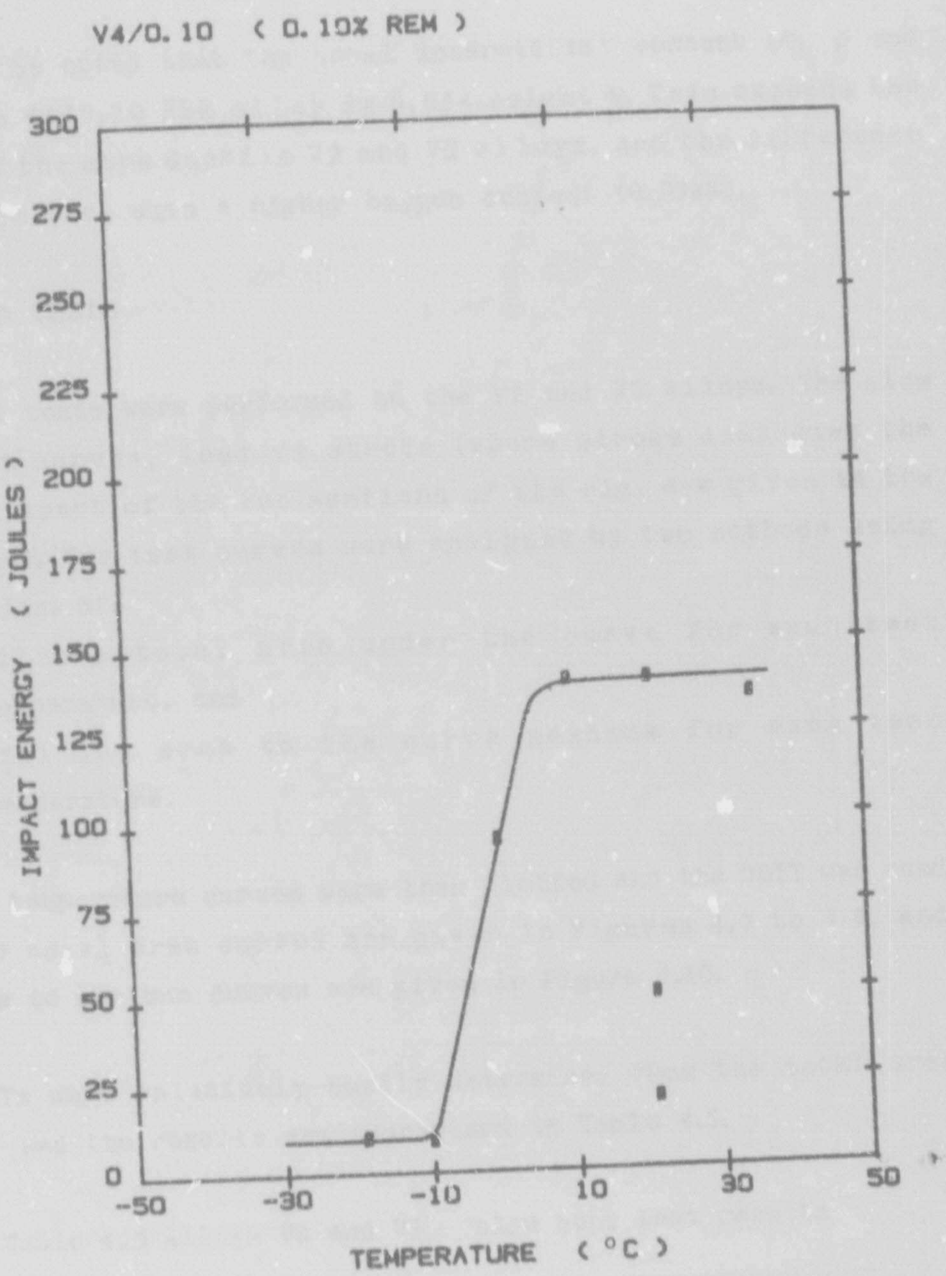

Figure 4.6 DBTT curve of the V4/0,10 alloy.

REM respectively. The upper shelf energy is considerably lower than those determined for all three of the V2 and V3 alloys.

It should be noted that the total interstitial content (C, O and N) for the V4/0,10 REM alloy is 0,054 weight %. This exceeds the values of the more ductile V2 and V3 alloys, and the difference again correlates with a higher oxygen content (0,038%).

4.3 SLOW BEND TESTS

Slow bend tests were performed on the V2 and V3 alloys. The slow bend test curves, load vs stroke (where stroke indicates the movement apart of the two sections of the rig) are given in the Appendix B. The test curves were analysed by two methods using calculations of:

(i) the total area under the curve for each test temperature, and

(ii) the area to the curve maximum for each test temperature.

Area vs temperature curves were then plotted and the DBTT was read off. The total area curves are given in Figures 4.7 to 4.9, and the area to maximum curves are given in Figure 4.10.

The DBTTs were relatively easily determined from the total area curves, and the results are summarised in Table 4.5.

Table 4.5 Alloys V2 and V3 - slow bend test results

Alloy	% REM added	DBTT	Transition temperature range (°C)
V2/1/2	0	10	30
V3/2	0,15	-10	20
V2/2/2	0,30	15	20

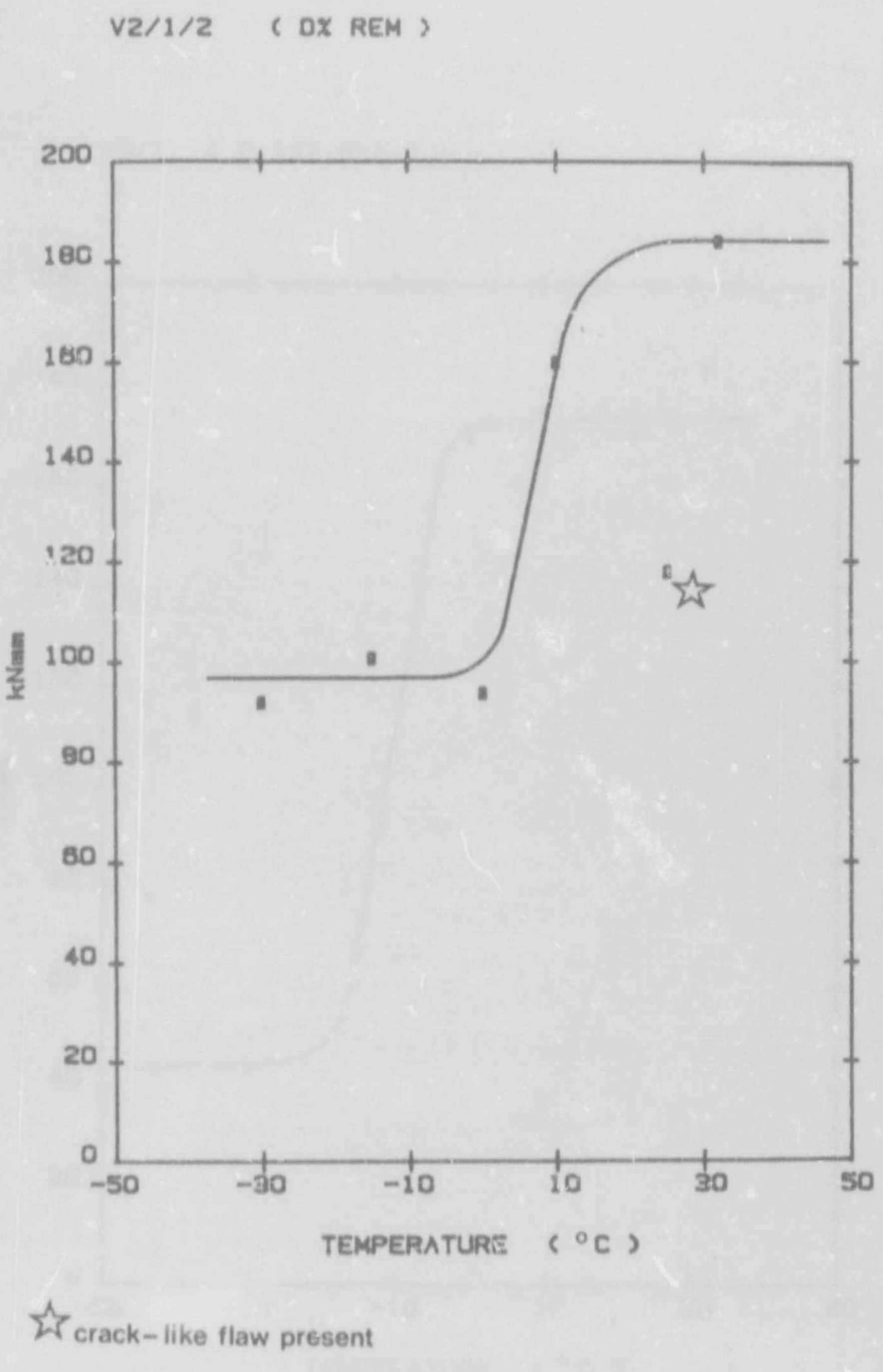

Figure 4.7 V2/1/2 Slow bend test DBTT.

(Total Area under curve)

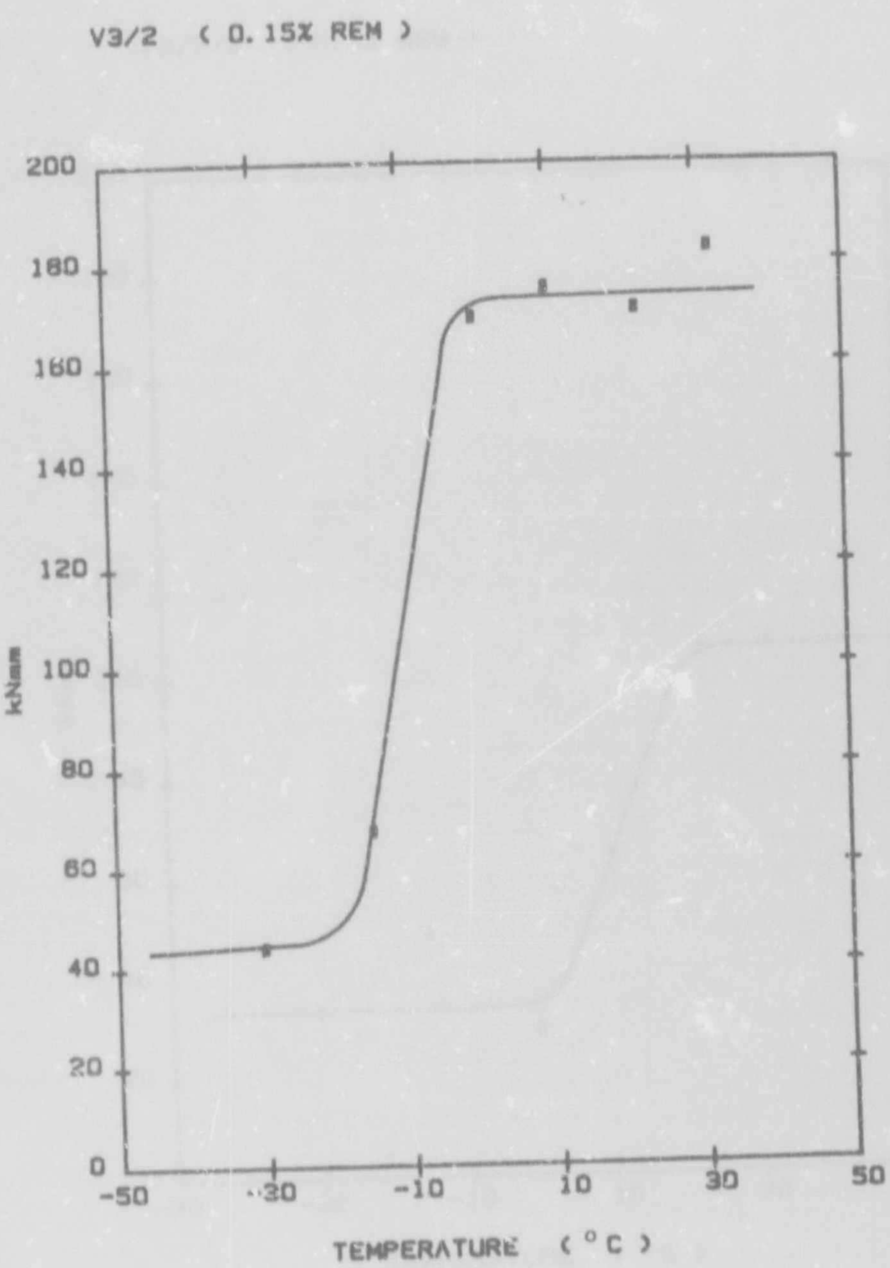

Figure 4.8 V3/2 Slow bend test DBTT.

(Total Area under curve)

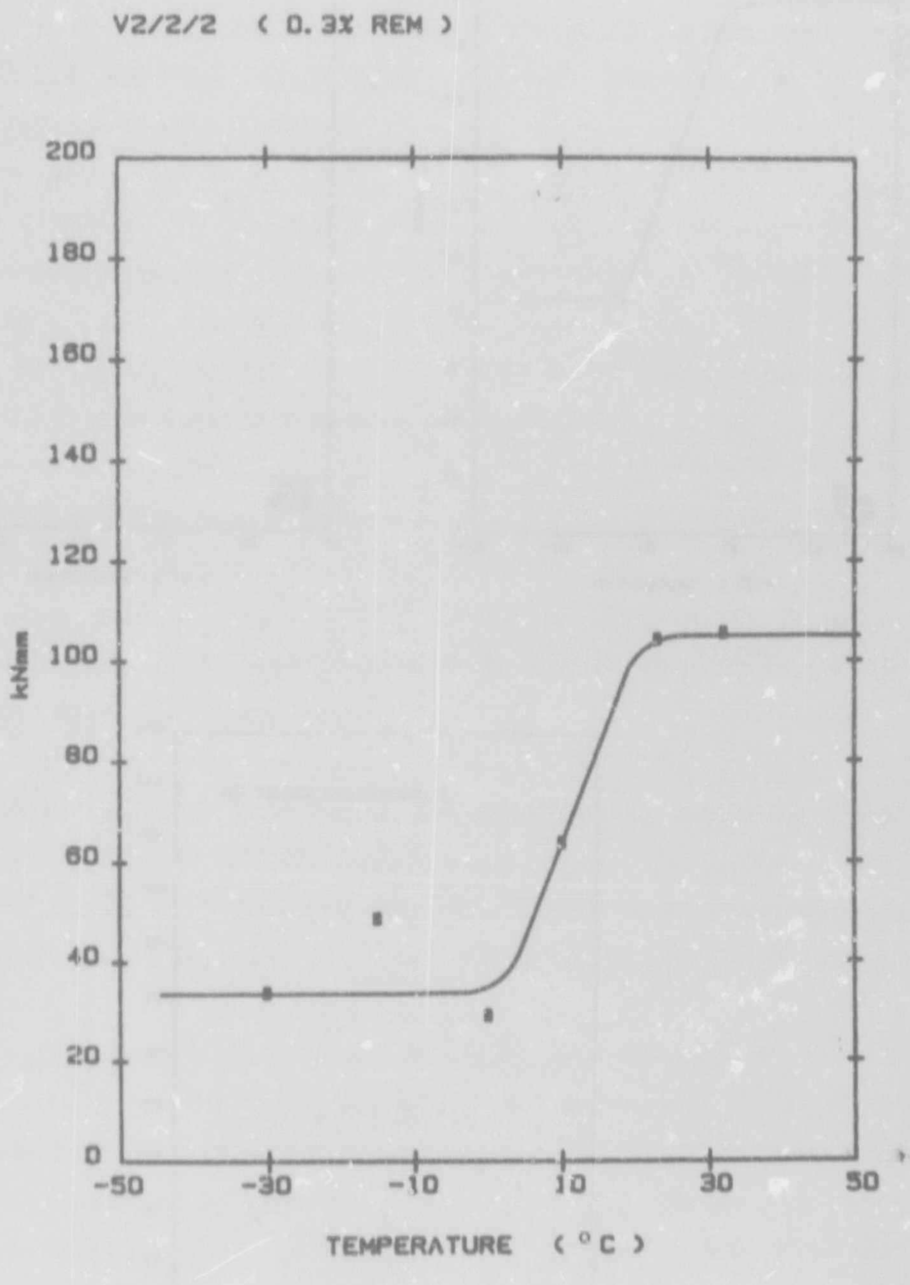

Figure 4.9 V2/2/2 slow bend test DBTT.

(Total Area under curve)

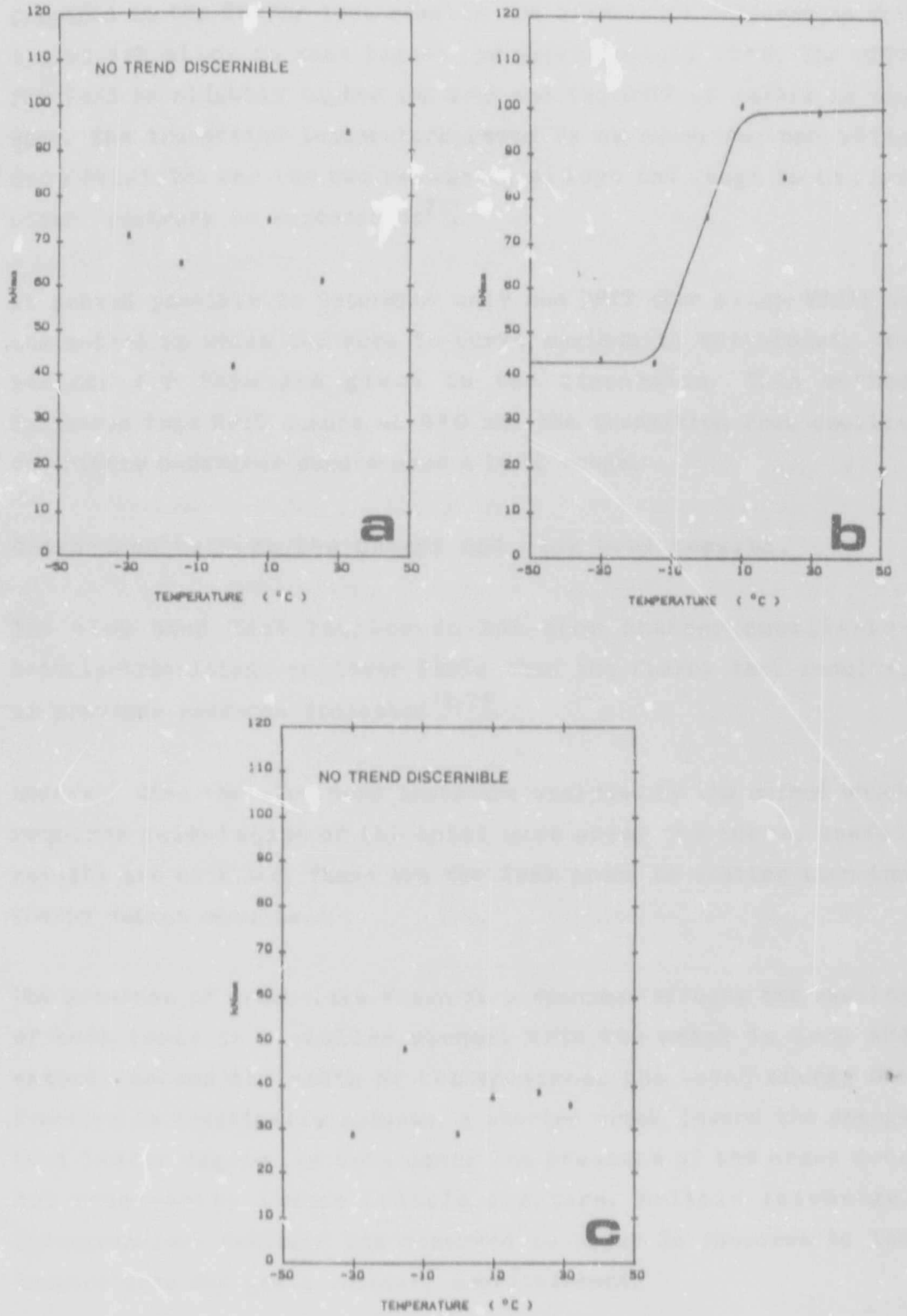

Figure 4.10 Slow bend test curves. (Area to curve maximum)

a) V2/1/2 (0%REM)

b) V3/2 (0,15%REM)

c) V2/2/2 (0,3%REM)

Compared to the Charpy test results the transition temperature for the V2/1/2 alloy is much higher, by approximately 20 °C. The DBTT for V3/2 is slightly higher (by 2 °C and the DBTT of V2/2/2 is the same. The transition temperature range is narrowed for one alloy only (V2/2/2). For the two remaining alloys the range is in fact wider, contrary to expectation[75].

It proved possible to determine only one DBTT (for alloy V3/2) by the method in which the area to curve maximum is calculated. The reasons for this are given in the discussion. This method indicates this DBTT occurs at 0 °C and the transition from ductile to brittle behaviour occurs over a 20 °C range.

4.3.1 Comparison between the Charpy and slow bend results.

The slow bend test results do not show sharper ductile-to-brittle-transitions or lower DBTTs than the Charpy test results, as previous research indicated[72,75].

However, when the slow bend tests are analysed by the method which requires calculation of the total area under the curve, useful results are obtained. These are far less prone to scatter than the Charpy impact results.

The presence of crack-like flaws in a specimen affects the results of both tests in a similar manner. When the crack is long and extends across the width of the specimen, the total energy for fracture is drastically reduced. A shorter crack lowers the energy to a lesser degree. In both cases the presence of the crack does not necessarily induce brittle fracture. Brittle (cleavage, intergranular) fracture was observed to occur in response to low temperatures and grain boundary embrittlement.

4.4 TENSILE TESTS

The tensile properties of the V2 and V3 and one of the V4 alloys were determined. An attempt was made to correlate the tensile test results to the amount of REM added.

4.4.1 The V2 and V3 alloys

The results of the tensile tests are summarised in Table 4.6 and presented in Figure 4.11.

The results show that the tensile properties for all three alloys are quite similar. The ultimate tensile strength shows a slight increase in value with increasing amounts of REM added. The yield strength on the other hand, appear to remain more or less constant for increasing amounts of REM addition. The ductility measures, % elongation and % reduction in area follow the opposite trend to that of the ultimate tensile strength.

Table 4.6 Tensile properties of the V2 and V3 alloys

Alloy	%REM added	σ_y (MPa)	UTS (MPa)	% elongation	%reduction in area
V2/1/2	0	396	540	32,5	57,5
V3/2	0,15	400	545	33	58,5
V2/2/2	0,30	397	559	38	68.5

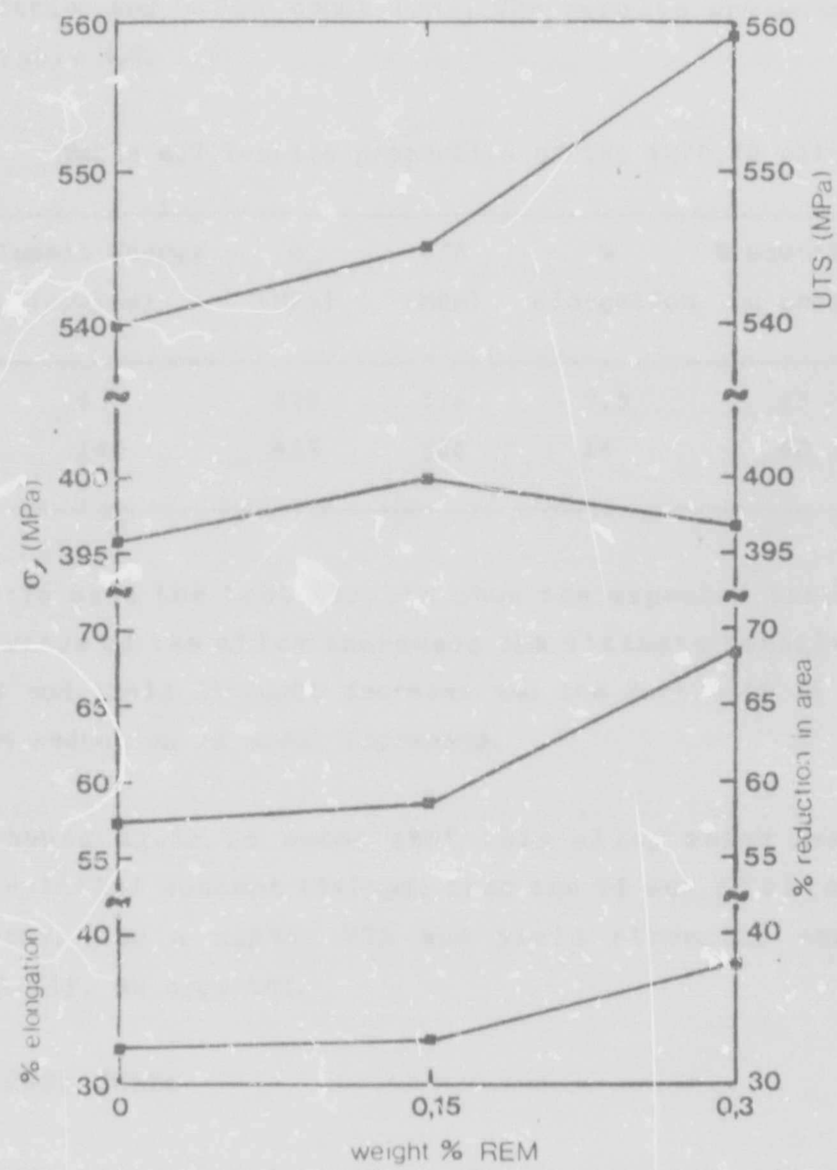

Figure 4.11 Tensile test results: σ_y, UTS, % elongation and % reduction in area versus weight % REM added.

4.4.2 The V4/0,10 alloy

The tensile properties of the V4/0,10 alloy were evaluated in the embrittled and 'tough' conditions. The results are summarised in the Table 4.7.

Table 4.7 Tensile properties of the V4/0,10 alloy

Impact Energy (Joules)	σ_y (MPa)	UTS (MPa)	% elongation	%reduction in area
6	479	576	7.5	35
140	457	566	16	60

In this case the test results show the expected trends. As the toughness of the alloy increases the ultimate tensile strength (UTS) and yield strength decrease and the ductility (% elongation and % reduction in area) increases.

It should again be noted that this alloy which has a higher interstitial content (540ppm) than the V2 and V3 alloys (380 to 450ppm), has a higher UTS and yield strength, and a lower ductility, as expected.

4.4 HARDNESS TESTS

In order to investigate the effects of composition and heat treatment on the mechanical properties of the Fe-40Cr alloy, hardness tests were carried out on some of the V1, V2, V3 and V4 alloys.

4.4.1 The V1 alloys

Hardness tests were performed on the V1 alloys used in the fabrication trials. The results are shown in Table 4.8.

Table 4.8 V1 alloys. The effect of annealing temperature hardness properties

Alloy	Annealing temperature(°C)	Annealing time (minutes)	Hardness (HV20)
V1/1	950	60	185
V1/2	1150	60	193

It can be seen that the higher annealing temperature produces a slight increase in hardness. These results are commensurate with those obtained in earlier studies documented in the literature survey (Section 2.1.5.1), in which it was also found that hardness increased and toughness decreased when the annealing temperature was raised.

4.4.2 The V2 and V3 alloys

The results of hardness tests carried out on these alloys are summarised in Table 4.9.

Table 4.9 V2 and V3 alloys. Hardness versus REM added to the melt

Alloy	REM added	Hardness (HV20)
V2/1/2	0	161
V3/2	0,15	161
V2/2/2	0,3	169

It appears that the hardness of an alloy is relatively insensitive to REM content.

4.4.3 The V4 alloys

Hardness tests were used to monitor the effects of annealing temperature and time on the mechanical properties of two of the V4 alloys. The results are shown in Table 4.10.

Table 4.10 The effect of annealing time and temperature on the hardness of two alloys, V4/0 and V4/0,1 with REM additions of 0 and 0,1 % by weight, respectively.

Alloy	% REM added	Annealing temperature (°C)	Annealing time (minutes)	Hardness (HV20)
V4/0	0	950	60	184
			90	183
			120	183
		1050	60	203
			90	200
			120	201
		1150	60	608
			90	213
			120	214
V4/0,1	0,1	950	60	185
			90	186
			120	184
		1050	60	202
			90	204
			120	206
		1150	60	210
			120	202
			150	203
			180	215

These results are presented graphically in Figure 4.12.

The values obtained show that for a given interstitial level (in this case about 550ppm) the hardness of an alloy is lower after annealing at 950 than at 1050 or 1150°C. This general trend is in agreement with the findings of previous studies on ferritic stainless steels (Section 2.1.5.1) and is due to solid solution hardening effects.

The relationship between impact energies (toughness) and absolute hardness values is, however, more complex than expected, since the 1150°C heat treatment corresponds to both a higher hardness and the restoration of ductility in the alloy V4/0,10. In this alloy, annealing at 1150°C produces hardness values of 210 and 215 HV after 1 and 3 hours respectively, and of about 203 HV at intermediate times. The higher values correspond to brittle behaviour whereas the lower values represent the condition in which toughness was recovered and ductile fracture observed.

In contrast, the V4/0 alloy annealed at 950, 1050 and 1150°C showed hardnesses ranging from 183 to 214 HV and all of these corresponded to brittle behaviour.

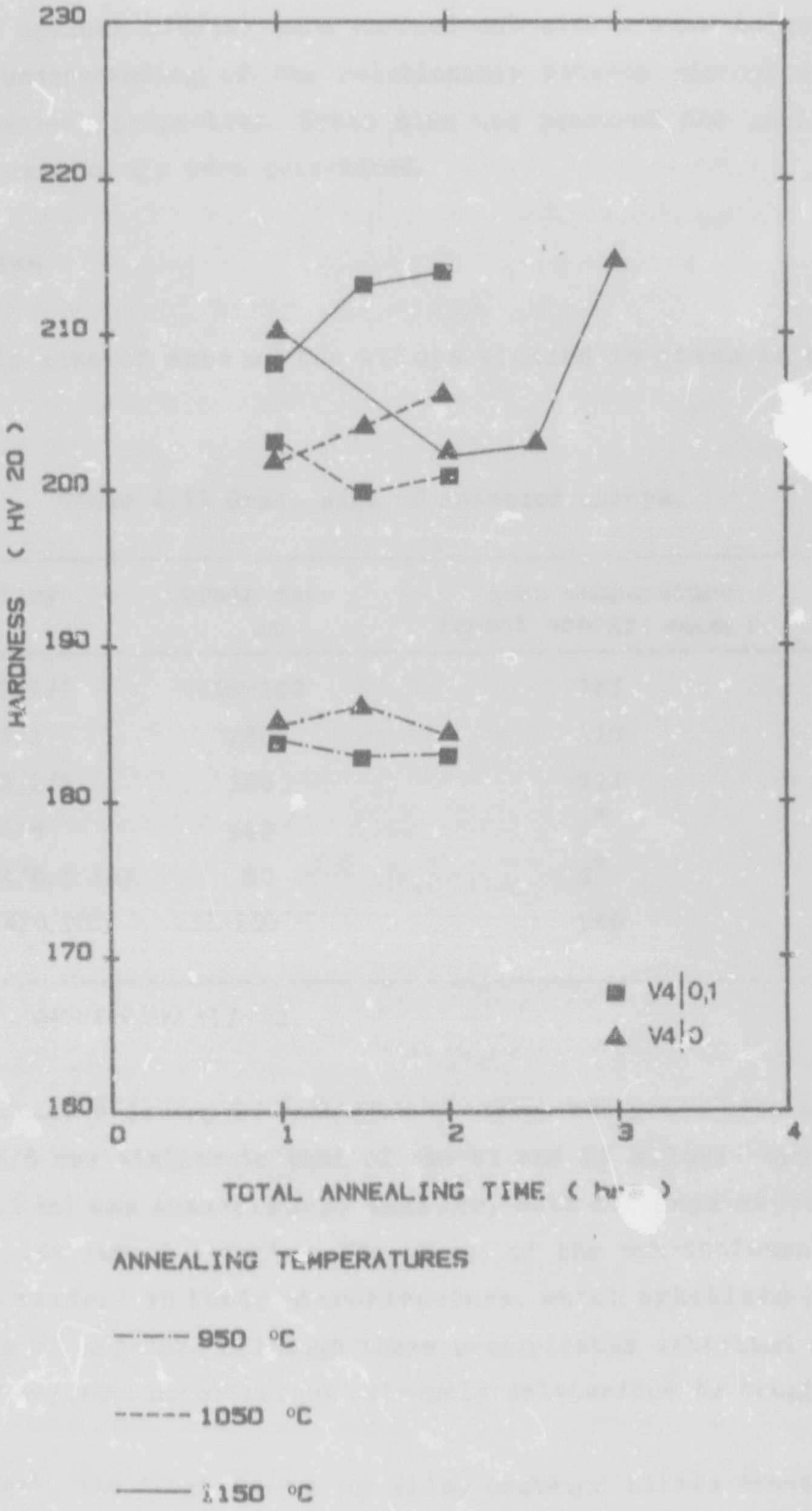

Figure 4.12 Hardness vs. annealing time for 950, 1050 and 1150 °C. V/0 and V4/0,10 alloys.