Rapidly quenched intermetallic compounds, TiAl and Al_3Ti

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Two types of molten intermetallic compounds, with stoichiometric compositions TiAl and Al_3Ti, are rapidly solidified at a cooling rate ranging from 10^4 to 10^5 K sec^{-1} using a melt-spinning method. Solidified specimens are analysed by X-ray diffractometry and observed by a high-voltage electron microscope and an analytical transmission electron microscope. Extra phases other than stoichiometric composition phases were not detected for either TiAl or Al_3Ti specimens by X-ray diffractometry. Both specimens have very small grains from 1 to 3 μm in diameter. TEM observation reveals that very fine precipitates (100 to 300 nm), which are not detected by X-ray diffractometry, are present within grains. They are Ti_3Al in the TiAl specimens, and aluminium in the Al_3Ti specimens, respectively. Electron micrographs of the Al_3Ti specimens show the presence of pair dislocations (super dislocations) and anti-phase boundaries. They are believed to have been formed at high temperature.

1. Introduction

The intermetallic compounds TiAl and Al_3Ti are expected to be potential candidate materials for high-temperature applications [1]. However, they have not been used practically because of a lack of room-temperature ductility [2]. In general, rapid solidification is advantageous for obtaining a very small grain size, or amorphous or metastable structures. Therefore, this method may be promising for improvement of the mechanical properties of intermetallic compounds.

TiAl, an $L1_0$ type intermetallic compound, belongs to a berthollide-type compound [3], which allows variation in its chemical composition within a single phase to some extent. On the other hand Al_3Ti, a DO_{22} type intermetallic compound, belongs to a daltonide-type compound [3], which does not allow any variation in chemical composition. Both compounds go through direct ordering from the liquid state.

Therefore it is interesting to examine the effect of rapid solidification on these two different types of compound. Even though they may not be solidified as an amorphous or a metastable state, they may have less precipitates and segregation of impurity compared with conventionally solidified materials. In the present study, solidified materials are pulverized and then sintered with the aim of obtaining a small grain size and a uniform matrix. The purpose of this study is to investigate the characteristics of rapidly solidified intermetallic compounds as candidate powder metallurgy materials for high-temperature applications.

2. Experimental procedure

TiAl was melted in an Al_2O_3 crucible and rapidly solidified from 1823 K by melt spinning on a 400 mm-diameter copper roll (melting point of TiAl, 1743 K). Similarly, Al_3Ti was melt-spun from 1723 K (melting point, 1613 K). The atmosphere was argon containing less than 20 p.p.m. O_2, and the cooling rate was estimated to be 10^4 to 10^5 K sec^{-1}. Solidified material in the form of flakes, about 3 cm in width and 60 to 100 μm thick, was analysed by X-ray diffractometry (XRD), and observed with a 1 MeV electron microscope and an analytical transmission electron microscope (TEM). Thin foils for TEM observation were prepared from the flakes by electrothinning in a Tenupol jet

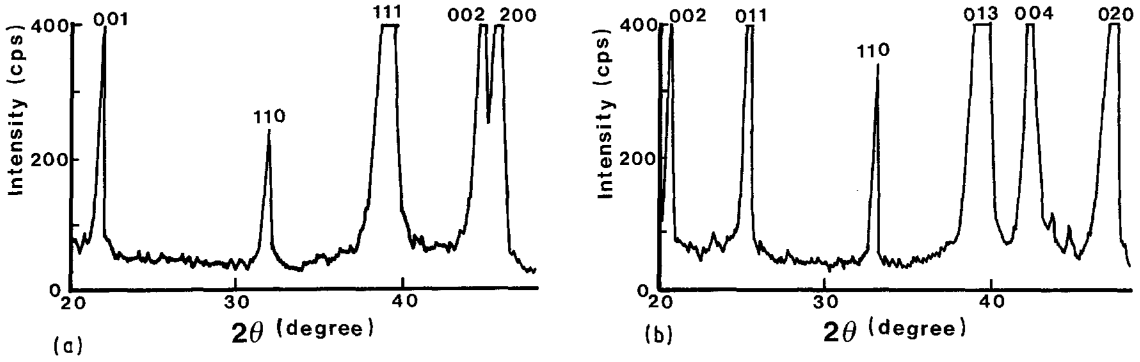

Figure 1 Results of X-ray diffractometry for (a) rapidly-quenched TiAl and (b) rapidly-quenched Al_3Ti.

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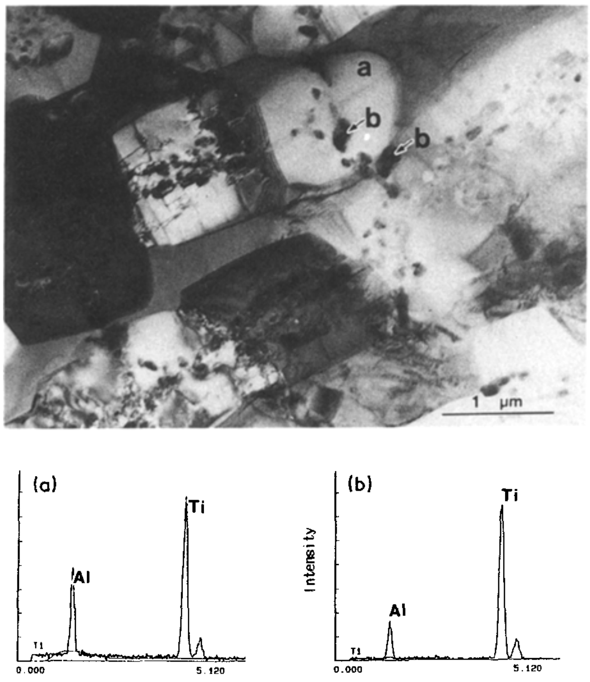

Figure 2 TEM image and EDS spectra of rapidly quenched TiAl. (a) TiAl; 53.28 at % Al–46.72 at % Ti. (b) Ti_3Al; 28.17 at % Al–71.83 at % Ti.

electropolisher at room temperature, with an electrolyte of 10% perchloric and 90% acetic acid.

3. Results and discussion

X-ray diffractometry indicates only the presence of stoichiometric compounds in both of the rapidly-solidified materials. The $L1_0$ structure (tetragonal, $a = 0.3999$ nm, $c = 0.4080$ nm, $c/a = 1.02$) for the TiAl specimen, and the DO_{22} structure (tetragonal,

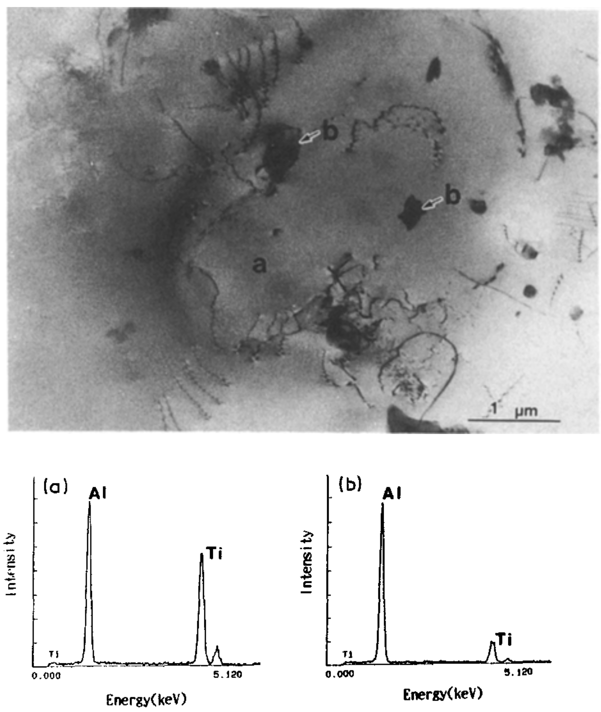

Figure 3 TEM image and EDS spectra of rapidly quenched Al_3Ti. (a) Al_3Ti; 72.52 at % Al–27.48 at % Ti. (b) Aluminium; 95.16 at % Al–4.84 at % Ti.

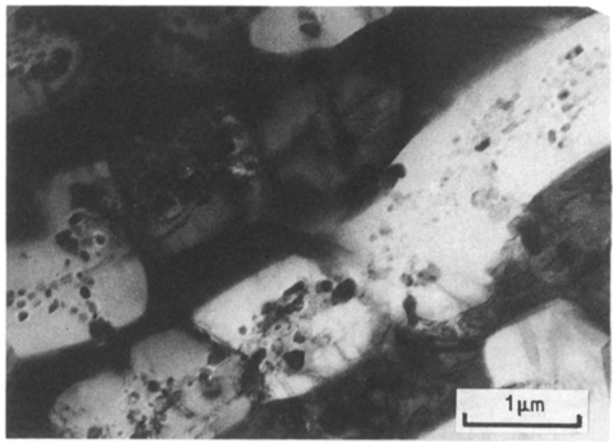

Figure 4 TEM image of rapidly solidified TiAl, the same area as Fig. 1 but with different contrast.

$a = 0.385$ nm, $c = 0.862$ nm) for the Al_3Ti specimen, were as shown in Fig. 1.

Figs 2 and 3 show TEM images and EDS spectra for rapidly solidified TiAl and Al_3Ti specimens, respectively. Both of the specimens have very small grain size, ranging from 1 to 3 μm in diameter. These micrographs show the presence of very fine precipitates (100 to 300 nm in diameter) in both of the specimens, although they were not detected by XRD. Precipitates in TiAl specimens were determined by EDS to be Ti_3Al, which has 28.2 at % Ti and 71.8 at % Al. The results of EDS analysis of fine precipitates in the Al_3Ti specimen shows the presence of a small titanium peak corresponding to 4.2 at % (Fig. 3b). This titanium peak is considered as a false peak which is generated by the surrounding matrix. Therefore the fine precipitates in the Al_3Ti specimen may be aluminium particles.

Fig. 4 shows a TEM image of a TiAl specimen, taken from the same area as in Fig. 2 but with different diffraction conditions. In this micrograph, several adjacent grains are seen to have similar contrast. This implies that similar crystallographic orientation may have been developed during solidification. Fig. 5 shows that some grains contain twins, which might have been formed during rapid solidification.

A heat treatment at 1273 K causes some structural

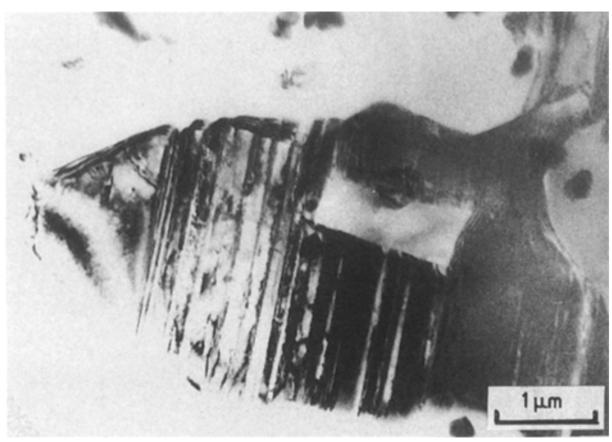

Figure 5 TEM image of rapidly solidified TiAl, showing twin structures in a grain.

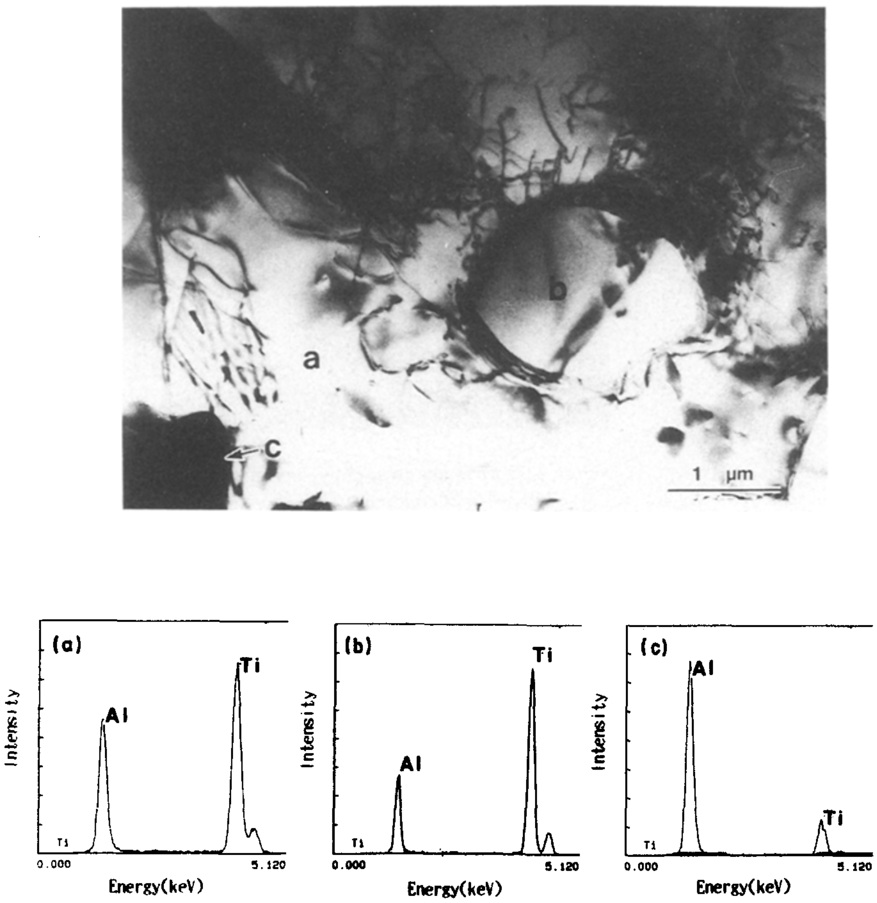

Figure 6 TEM image and EDS spectra of rapidly quenched TiAl, annealed at 1273 K. (a) 56.56 wt % Al–43.44 wt % Ti. (b) 42.85 wt % Al–57.15 wt % Ti. (c) 91.04 wt % Al–8.96 wt % Ti.

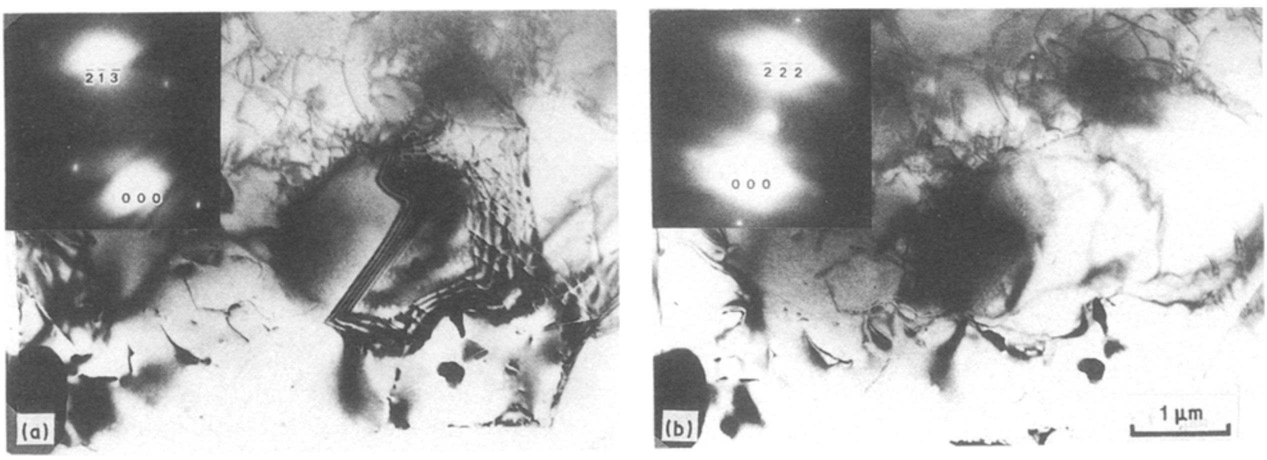

Figure 7 TEM image of rapidly quenched TiAl, annealed at 1273 K, showing strain between matrix and a precipitate.

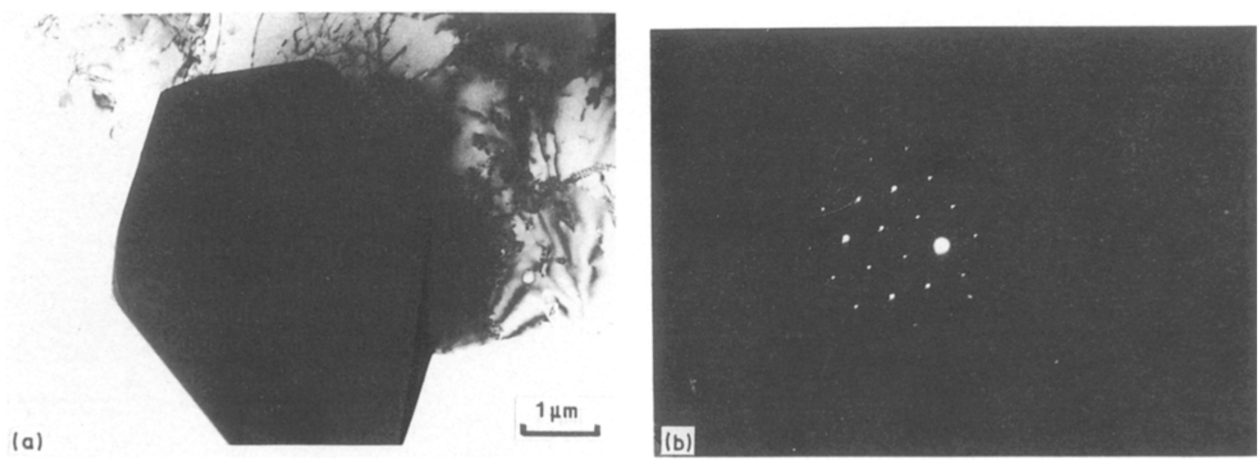

Figure 8 TEM image of rapidly quenched TiAl, annealed at 1273 K, showing Ti_3Al as a second phase.

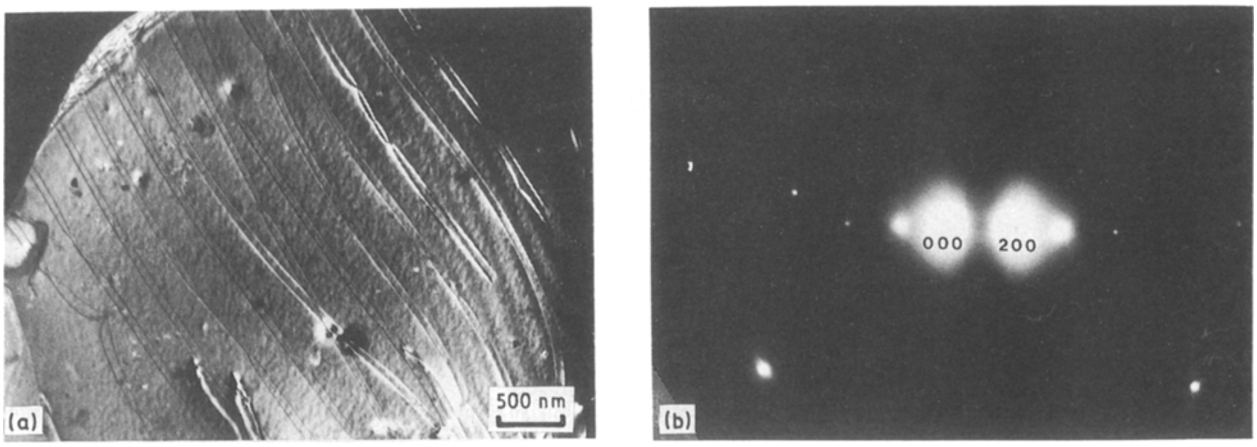

Figure 9 TEM image of rapidly quenched Al_3Ti, showing super dislocation separated into two partials.

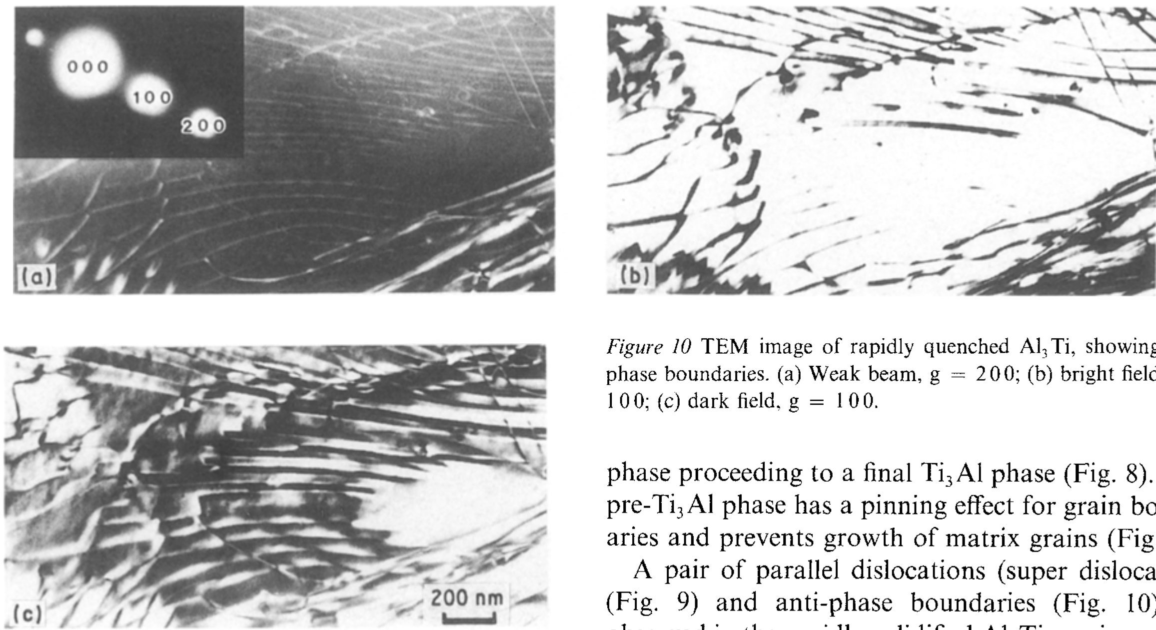

Figure 10 TEM image of rapidly quenched Al_3Ti, showing anti-phase boundaries. (a) Weak beam, g = 2 0 0; (b) bright field, g = 1 0 0; (c) dark field, g = 1 0 0.

changes in rapidly solidified TiAl, which are shown in Figs 6, 7 and 8. Fig. 6 shows a TEM image with EDS spectra obtained from TiAl, which was rapidly solidified and annealed at 1273 K for 24 h. The analysis reveals three phases, namely the matrix (a), titanium-rich phase (b), and aluminium-rich phase (c). The titanium-rich phase is accompanied by strain fields at the interface (Fig. 7) and is thought to be a metastable phase proceeding to a final Ti_3Al phase (Fig. 8). This pre-Ti_3Al phase has a pinning effect for grain boundaries and prevents growth of matrix grains (Fig. 7).

A pair of parallel dislocations (super dislocation) (Fig. 9) and anti-phase boundaries (Fig. 10) are observed in the rapidly solidified Al_3Ti specimen. It is concluded from the diffraction condition that these super dislocations lie on a (0 0 1) plane, which is considered to have the lowest anti-phase boundary energies at high temperature in this system [4]. These super dislocations seem to be pinned down by fine aluminium precipitates as shown in Fig. 11.

4. Conclusions

The results for the two types of rapidly quenched

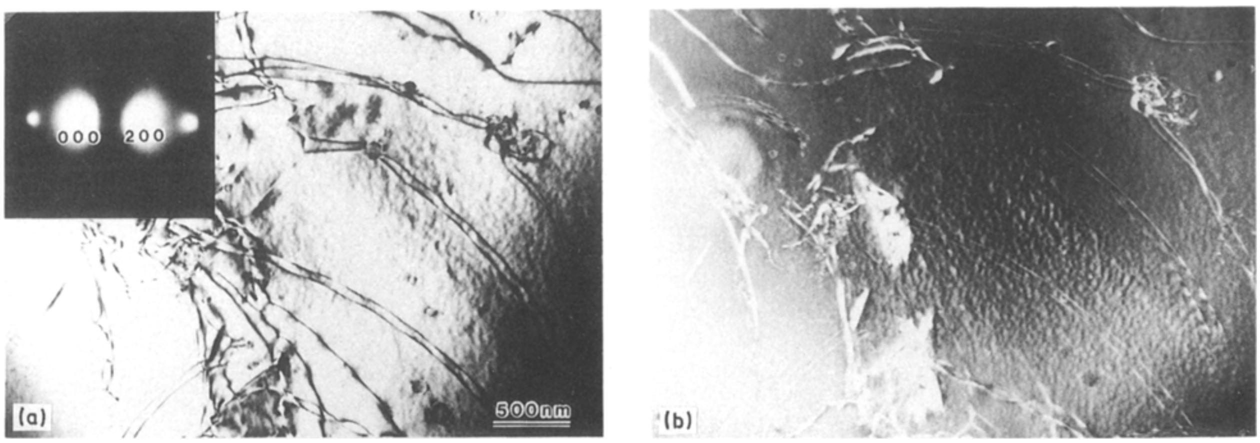

Figure 11 TEM image of rapidly quenched Al_3Ti, showing superdislocations pinned by precipitates.

intermetallic compounds, TiAl and Al_3Ti, are summarized as follows.

1. Macroscopically uniform structures having stoichiometric compositions are obtained by rapid solidification for both of the melt-spun specimens.

2. The grain sizes of both specimens are very small and range from 1 to 3 μm in diameter.

3. TEM observations reveal that fine precipitates (100 to 300 nm) are present within grains, which are not detectable by XRD. Precipitates are Ti_3Al in the TiAl specimen and aluminium in the Al_3Ti specimen.

4. The Al_3Ti specimen shows pair-line images of dislocations (super-dislocation) and antiphase boundaries. They are believed to be formed at high temperatures.

References

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