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V. Vitek, D. P. Pope, D.E. Luzzi

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Single crystals of six different compositions were grown, five of Al_3 Ti + Fe and one of Al_3 TI + Cr. The flow stress was measured as a function of temperature, orientation and composition. Al_2Ti precipitates are the major strengthening phase and form platelets on the {100} planes of the $L1_2$ matrix. Deformation occurs primarily by <110> {111} slip, with SISF dissociation at low temperatures and by APB dissociation at high temperatures. Simulations of dislocation cores using many body potentials have revealed a variety of core configurations in the $L1_2$ and DO_{22} form, all of which are sessile, and one of which provides a twinning mechanism.

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V. Vitek, D.P. Pope, D.E. Luzzi

University of Pennsylvania
Philadelphia, PA 19104

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PLASTICITY OF SINGLE CRYSTALLINE AL_3X INTERMETALLIC COMPOUNDS

V. Vitek, D. P. Pope and D. E. Luzzi
Department of Materials Science and Engineering
University of Pennsylvania,Philadelphia, PA 19104

Deformation Behavior of $L1_2$ Al_3Ti

In this final report we summarize the results of this program but concentrate only on those portions which were not described in the two progress reports dated 7/3/90 and 4/6/91.

Introduction

In this program, single crystalline Al_3Ti was produced in the $L1_2$ form by additions of Fe or Cr and the deformation behavior was studied. It was both an experimental and theoretical program in which the causes for the extreme brittleness of this alloy was investigated. In the course of this investigation, a number of different studies were carried out. These were:

1. Growth of single crystalline alloys of 6 different compositions, see Table 1.
2. Measurements of CRSS and ductility as a function of strain rate and temperature.
3. Slip trace analyses.
4. TEM measurements of dislocation Burgers vectors and dissociations.
5. Identification of second phases.
6. Development of suitable many-body potentials for the DO_{22} structure.
7. Fault energies and core dissociations were calculated using these potentials.
8. A new model for twinning in the DO_{22} structure was developed.
9. Core phenomena controlling the plastic flow in Al_3Ti in the $L1_2$ form were studied.
10. A new, more complete model describing anomalous flow in the $L1_2$ compounds was developed.

Table I: Compositions of Single Crystals Grown as Part of This Study

Designation	Al (%)	Ti (%)	Fe (%)	Cr (%)
S1	67.0	25.0	8.0	-
S2	65.2	26.6	8.2	-
S3	66.8	27.4	5.8	-
S4	64.8	28.4	6.8	-
S5	63.7	28.7	7.6	-
S6	67.0	25.0	-	8.0

Microstructure

As will be discussed later in this report, the existence of second phases have a large effect on the mechanical properties of Al_3Ti and are therefore discussed in some detail.

Ti_2NAl

Early in this study, a Ti-rich phase with composition identified as being near $Ti_{66.7}Al_{33.3}$ was observed in single crystalline $Al_{65.2}Ti_{26.6}Fe_{8.2}$ and $Al_{66.8}Ti_{27.4}Fe_{5.8}$. The shape of the phase is of two types when viewed in cross section: one has a more or less rectangular shape of typical size around 25-40 μm, and the other forms narrow ribbons 0.1-1.5 mm long and about 10 μm thick. The formation of a Ti-rich phase in the Al-rich matrix was puzzling, since it did not seem to be relevant to the overall composition of the compounds, and was hard to understand based on considerations of the solidification sequence. Furthermore, the phase is not reported in the Al-Ti-Fe isotherms measured at 1000 and 800°C (Markiv et al 1973, Siebold 1981, and Mazdyasni et al 1989).

However, a so-called "Ti_2Al" phase was previously found in γ-TiAl, and has been studied by a number of groups (Loiseau and Lasalmonie 1983_a, Loiseau et al 1983_b, Loiseau and Lasalmonie 1984, and Kaufman et al 1986). According to Loiseau and coworkers, the phase only exists in Al-rich TiAl, and is hexagonal with lattice parameters $a = 3.0$ Å and $c = 14.0$ Å. Kaufman et al (1986) studied more recently this phase again using TEM. Consistent with the work of Loiseau and coworkers, they found that the phase only exists in Al-rich TiAl, and the lattice parameters are $a = 3.04$ Å and $c = 13.89$ Å. After analyzing the convergent electron beam diffraction patterns, they determined that the space group of the phase is $P6_3/mmc$. Making use of EELS, they observed an additional edge corresponding to that of N in the spectrum. Based on

these results, they concluded that the so-called "Ti_2Al" is actually Ti_2NAl which was previously known to have the same crystal structure as that of Cr_2CAl. The reason why the second phase only exist in the Al-rich but not the Ti-rich TiAl, as explained by these workers, is that the Al-rich compounds are single γ-phase materials, while the Ti-rich compounds are two-phase ($\alpha_2 + \gamma$) materials, and in the two-phase materials the N partitions to the α_2 where the solubility of the element is much greater and remains in solution.

Enlightened by these experiment results, it was decided to investigate this phase further. The composition of the Ti-rich phase in our samples was re-identified using the EDAX technique as before but with the beryllium window removed so that light elements could be seen. Indeed, the phase was found to contain nitrogen, and the Ti-rich precipitates contain much more N than does the matrix. Unfortunately, N could not be quantitatively analyzed using x-ray intensities with the same confidence as elements heavier than sodium. Uncertainty in mass absorption coefficients is the main hurdle to using the standard ZAF correction schemes.

In order to determine whether the crystal structure of the Ti-rich phase seen in the $L1_2$ Al_3Ti is the same as that of the Ti_2NAl, a specially prepared powder specimen containing a large volume fraction of the precipitates was examined using a diffractometer, and the obtained spectrum was compared with the computer simulated spectrum of the Ti_2NAl. For the simulation, the previously-determined space group and atom positions were used as inputs. The simulated spectrum best matched the experimental one when the lattice parameters were chosen to be a = 2.995 Å and c = 13.61Å, which differs only slightly from the ones determined by previous workers.

Since the Ti-rich phase is actually the Ti_2NAl rather than Ti_2Al, it should not be surprising that the phase is absent from the Al-Ti-Fe isotherms. The nitrogen very likely came from the Ti starting material. During casting, a small amount of Ti might have formed a metastable Ti-rich phase containing N. When the cast material was remelted during subsequent crystal growth, Al atoms diffused into these particles and formed the Ti_2NAl. An explanation like this seems to agree well with the experimental observations that the Ti_2NAl seen in the S3 single crystal (the starting material of which was Ti powder) were exclusively small particles of rectangular shape, while in other single crystals (prepared from Ti rods) the majority of the precipitates seen were in the shape of relatively long and thin sheets.

Al_2Ti

A second phase, the composition $Al_{65.8}Ti_{33.7}Fe_{0.5}$, was first observed in the S3 composition single crystalline compound. The phase forms planar precipitates on the cube planes of the $L1_2$ matrix, as determined by a two-surface analysis. It was later found that the phase exists in almost all the compounds used for this study, except for those containing low Ti (<25 at. %).

In the Al-Ti binary system, a metastable Al_2Ti phase with very narrow solubility range exists at temperatures below about 1200°C. The phase is body centered tetragonal with lattice parameters a = 3.976 Å and c = 24.36 Å. The structure of the phase can be considered to be formed from six nearly fcc cubes stacked up along the c-axis, as shown in Fig. 1. In the ternary Al-Ti-Fe system, however, the existence of Al_2Ti is still a controversial topic. Markiv et al (1973) and Mazdiyasni et al (1989) identified Al_2Ti in equilibrium with the $L1_2$ phase, but this conflicts with the work of Siebold (1981). Even in the work in which the Al_2Ti was identified, detailed information, such as the solvus temperature, solubility of the ternary element, and the orientation relation between the Al_2Ti and the $L1_2$ are not given. In view of the important effects of the phase on the mechanical properties of the $L1_2$ compounds, as is discussed in a later section, a detailed study on the phase has been carried out.

X-ray powder diffraction spectra obtained from an S3 specimen showed, in addition to the intensity peaks from $L1_2$ matrix, a number of peaks from the Al_2Ti phase. Since the precipitates contain only 0.5 at% of Fe, the crystal structure of the phase is likely the same as that of the binary Al_2Ti. To prove this, computer simulations similar to the ones done previously for the Ti_2NAl were conducted and compared to the x-ray spectra. The experimental and simulated spectra matched quite well. The lattice parameters of the phase thus determined are a = 3.951 Å and c = 24.20 Å. The value of the a-parameter is very close to that of the matrix lattice parameter (3.938Å), differing only by about 0.3%. In comparison with the binary Al_2Ti, the small amount of Fe has significantly changed the a-parameter, making it possible for the phase to grow coherently on the cube planes of the matrix. As a matter of fact, all the intensity peaks of the $L1_2$ matrix overlap with the corresponding peaks of the Al_2Ti.

DTA measurements and in-situ heating experiments in a TEM were also performed. In the DTA an exothermal process was seen to occur between 600°C and about 950°C, corresponding to a continuous dissolution of the Al_2Ti. A thin foil prepared from an S3 specimen was heated in a TEM to 180, 550, 600, 650, 700, 800, 900 and 1000°C, and the temperature was then held for an hour or longer, to reach equilibrium. It was observed that the precipitates continuously dissolved

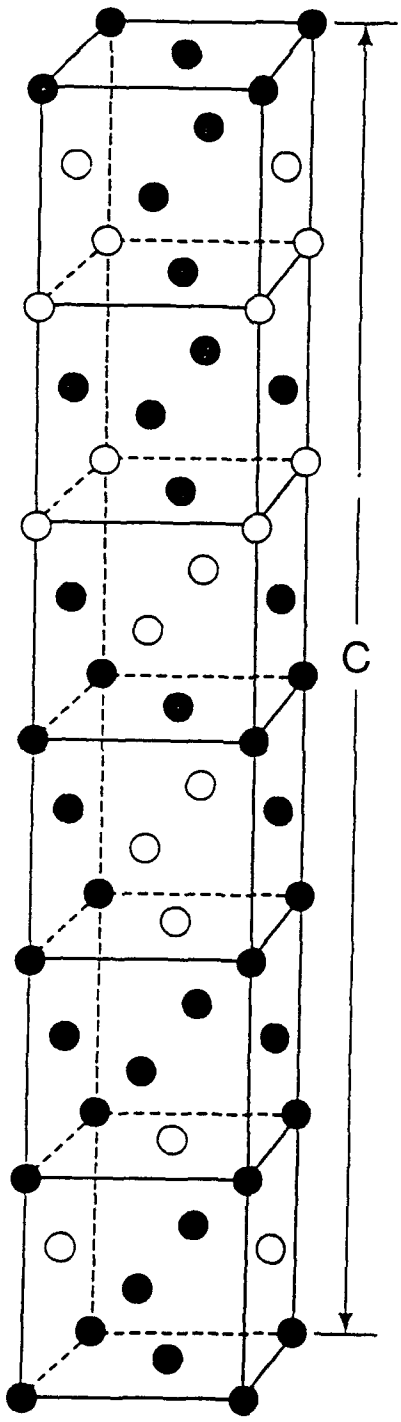


Fig. 1. **The crystal structure of Al_2Ti.**

starting at 650°C, and that the majority of the precipitates were completely dissolved at 900°C. Based on these results, it is clear that the size of the $L1_2$ phase field changes with temperature between 900 and 650°C.

As the result of the wide range of temperature over which the Al_2Ti forms, the precipitates vary greatly in size. During slow cooling after annealing, the first precipitates tend to be coarse. At relatively lower temperatures, the diffusivities are substantially reduced, the diffusion distance much shorter, and only fine precipitates can be formed. The size of the precipitates has also been found to depend strongly on a cooling rate. It is generally true that the faster the cooling rate, the finer the precipitates. At very high cooling rates, the Al_2Ti may not form or form at such a fine scale that it cannot be seen by optical microscopy.

In order to confirm directly the crystallographic relationship between the Al_2Ti and the $L1_2$ matrix, a few thin foils with planes close to the (100) and (110) planes of the matrix were prepared from the S3 crystal, and examined by TEM. Fig. 2 (a) and (b) show a bright field image and an electron diffraction pattern from one platelet taken along the [100] direction. The Al_2Ti precipitates seen in the bright field image are on the (010) and (001) planes. Most of the diffraction spots satisfy the reflection conditions of the crystal structure of Al_2Ti, but some double diffractions of the $(002n)_p$ type (where n=1, 3, 5 ..., and p stands for precipitate) also exist. The lattice parameter mismatch $(c - 6a)/6a$ (where c is the c-parameter of the Al_2Ti and a is the lattice parameter of the matrix) is 2.4%, which agrees very well with that determined directly from the lattice parameters obtained from x-ray diffraction data, 2.95%. Thus it is seen that the Al_2Ti precipitates form a planar array of platelets having a large range of sizes on the {001} planes of the $L1_2$ matrix. This distribution of precipitates results in very significant strengthening of the material.

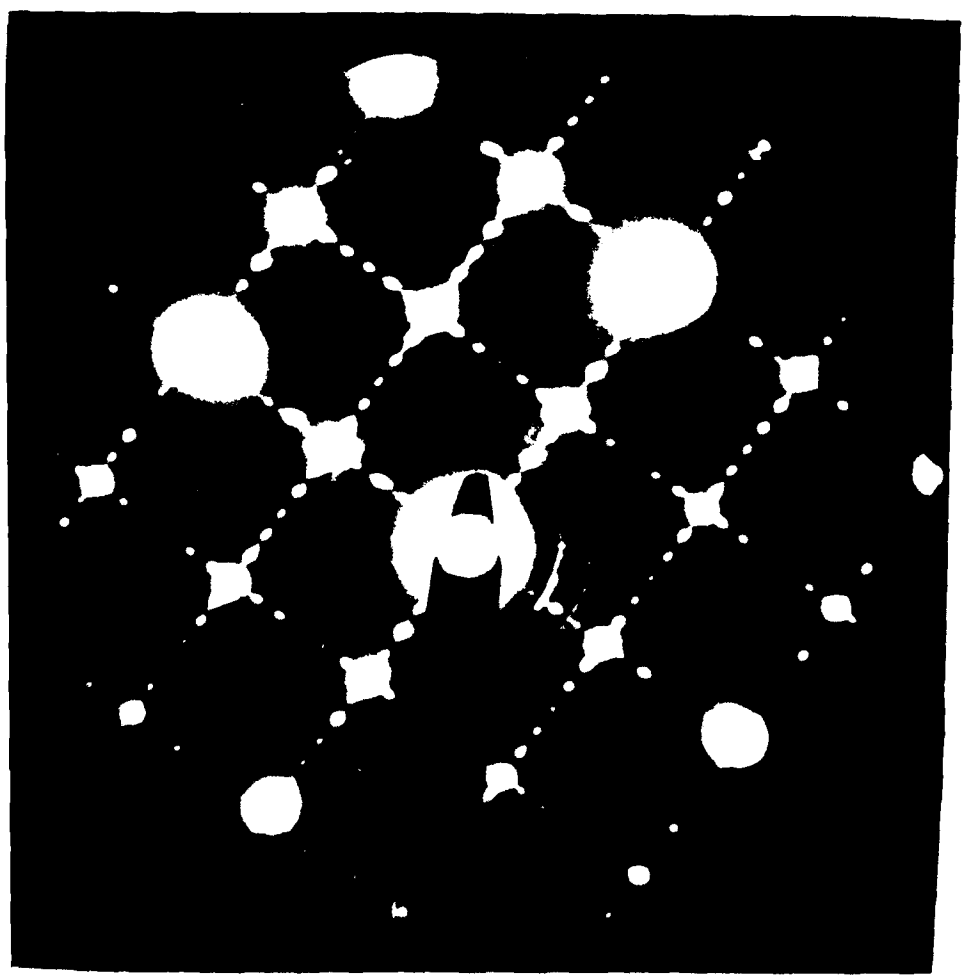
Deformation and Fracture of $L1_2$ Al_3Ti

As discussed in the two earlier annual reports, single crystals were produced by the optical imaging floating zone technique and by the Bridgman method. Compression tests were performed on parallelopipeds approximately 3x3x5mm of various orientations and at a range of temperatures ranging from 77 to 1300K. The operating slip planes were identified using Nomarski interference contrast optical microscopy.

Preliminary experiments on polycrystalline $Al_{67}Fe_9Ti_{24}$ showed that there is only a relatively small strain rate dependence of the flow strength and that the compressive ductility is largely the result of microcracking (Wu et al. 1990). There is no ductility observed in bending tests at room temperature and failure occurs by transgranular cleavage.

(a)

Bright field image. Incident beam along [100]. Single crystalline $Al_{66.8}Ti_{27.4}Fe5.8$



(b)

An electron diffraction pattern taken from the same area as the one above. Incident beam along [100].

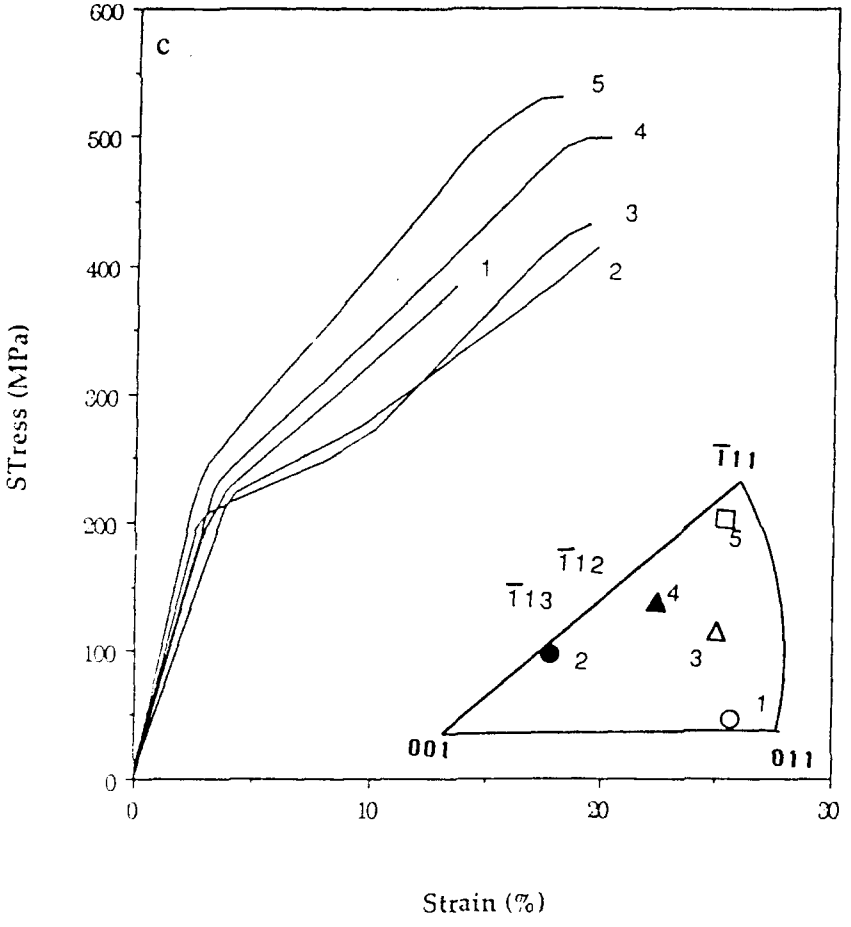
Fig. 2. **The Al_2Ti precipates.**

Experiments on single crystalline samples (Wu et al, 1990), showed that there can be large differences in CRSS, depending on composition (higher Ti content implies higher CRSS), that octahedral slip is observed in all samples but cube slip is observed at high temperatures in strong crystals orientated near [111], second phase $AlTi_3$ (later identified as Ti_2NAl) ribbons are preferred fracture paths, and that cleavage occurs on many planes, including (110), (001), (013) and (111) planes. The main results obtained since the second annual report dated 4/6/91 are the following:

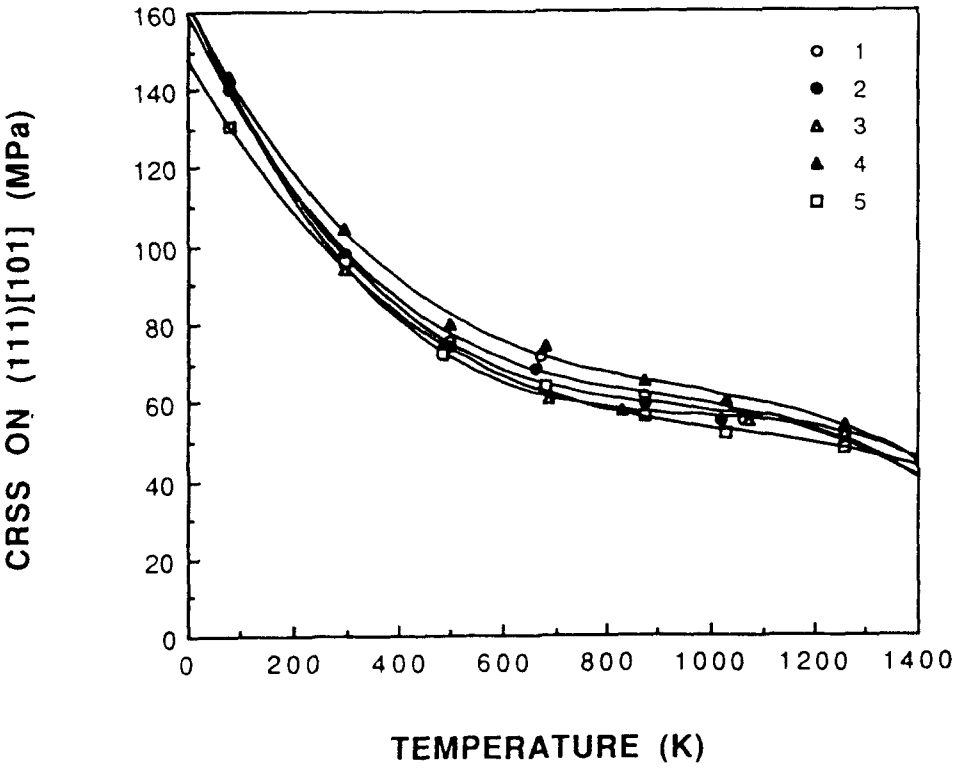
1. The large increases in strength observed with increasing Ti content are due to a fine distribution of Al_2Ti platelets on (001) planes of the $L1_2$ matrix, as discussed in the previous section.
2. The "$AlTi_3$" phase, seen in either rectangular blocky form or in ribbon-like form and which is the preferred fracture path in the latter form, is actually Ti_2NAl.
3. When there is no second phase Al_2Ti in the structure, or when the volume fracture of that phase is small, slip occurs only on the octahedral system, and the CRSS is independent of orientation and composition (but very dependent on temperature). Fig. 3 shows stress-strain curves and CRSS vs temperature curves for Cr-modified $L1_2$ ($Al_{67}Ti_{25}Cr_8$) single crystals. The curves of CRSS vs. temperature for Fe modified crystals (this study) and Mn-modified crystals (unpublished results of S. Brown from AFOSR-supported work on this laboratory) are identical to those in Fig. 3.

Transmission Electron Microscopy

In the last progress report, dated 4/6/91, results on Fe-modified $L1_2Al_3Ti$ were reported showing that the nature of the dislocation dissociation in this material changes with deformation temperature. Dislocations produced by deformation at room temperature lie on {111} planes and dissociate into two 1/3<112> superpartial dislocations separated by SISF; however, dislocations produced by deformation at 600°C lie on {100} planes and dissociate into two 1/2<110> superpartials separated by APB. More samples have been examined using weak beam TEM, including Cr-modified single crystals ($Al_{67}Ti_{25}Cr8$). In this material deformation at 77K results in 1/3<112> superpartials separated by SISF on {111} planes, the same as described above for the Fe-modified material deformed at room temperature. Deformation of the Cr-modified single crystals at 750°C results in 1/2<110> superpatials separated by APB, the same as in the Fe modified material at 600°C, but the plane of dissociation is {111}, not {100} as for the Fe-modified material. The slight differences between the two results suggests that the exact nature of the dissociation depends on the composition of the alloy, but there seems to be no doubt about the fact that an SISF-type dissociation occurs at low temperatures and an APB-type dissociation occurs



(a)



(b)

Fig. 3. (a) Stress-strain curves at room temperature and (b) temperature dependence of the CRSS for {111}<110> slip in $Al_{67}Ti_{25}Cr_8$ single crystals.

at higher temperatures. This observation, combined with the fact that the core of the SISF-type dissociated dislocation is highly nonplanar, explains the strong temperature dependence of the CRSS for {111} slip at low temperatures and also explains the plateau at higher temperatures (where APB-type splitting occurs which allows these dislocations to be planar and move relatively easily without thermal activation).

Theoretical Analysis of Dislocation Core Structures

Al_3Ti in $L1_2$ Form

As discussed in the last annual report, a model, many-body potential was developed which leads to a very high APB energy and an unstable CSF on {111} planes. For such a potential splitting into the 1/3<112> superpartials separated by SISF on {111} planes is favored.The atomistic calculations performed using this potential show that the 1/3<112> superpartials possess sessile cores. The consequence of this core structure is then the rapid increase of the flow stress with decreasing temperature at low temperatures (see also Tichy, Vitek and Pope 1986). This explains, as also discussed in the previous section, the low temperature yield behavior of Al_3Ti in $L1_2$ form. However, the present atomistic studies do not explain the transition towards APB splitting at higher temperatures and further studies of this phenomena are needed to fully understand the plastic behavior of Al_3Ti in the $L1_2$ form.

In addition to the above, a more complete theory of the anomalous flow stress, which is observed in Ni_3Al and other $L1_2$ compounds with high APB energy and metastable CSF, has been developed. It depends on two new concepts: (1) a pinning process involving an effect of the internal stresses due to ternary elements and (2) a thermally-assisted unpinning process. A comprehensive model for the pinning mechanism was developed by Paidar, Pope and Vitek (Paidar et al 1984), hereafter referred to as PPV. This model is based on earlier considerations of the cross-slip of screw dislocations in $L1_2$ compounds (Kear et al 1962, Takeuchi et al 1973) and on the results of atomistic calculations of the dislocation cores (Yamaguchi et al 1982, Vitek et al 1991) which suggest that when the antiphase boundary (APB) is on the (111) plane the core of the 1/2[$\bar{1}$01] superpartial may possess two alternate configurations: a higher energy glissile configuration which can be regarded as splitting into two Shockley partials bounding a ribbon of complex stacking fault (CSF) on the (111) plane, and a lower energy sessile configuration which corresponds to the formation of a narrow ribbon of APB on the (010) plane with the superpartial split into two Shockley partials bounding a ribbon of CSF on the (1$\bar{1}$1) plane. The PPV model was very successful in explaining the orientation dependence of the yield stress and the T/C asymmetry. It was demonstrated that the T/C asymmetry was caused by stress-induced narrowing

on $(1\bar{1}1)$ planes, the so-called "Escaig effect" (Vitek 1985, Escaig 1979, Bonneville et al 1979). This model predicted that the orientations for which there is no T/C assymmetry lie on a line which deviates significantly from the [012]-[$\bar{1}$13] line, in the standard triangle [001]-[011]-[$\bar{1}$11], towards the [001] corner. It also predicted that the maximum T/C asymmetry should occur for orientations close to [011].

While the T/C asymmetry observed in ternary compounds of Ni_3Al [20,21] is in very good agreement with these predictions, recent observations on binary Ni_3Al [22] and Ni_3Ga [23] suggest certain discrepancies. In particular, the orientations for which the T/C asymmetry vanishes have been found to lie very close to the [012]-[$\bar{1}$13] line in both these binary compounds and the T/C asymmetry is approximately the same for orientations near [001] and [011]. A modified incorporation of the "Escaig effect" involving shear stresses perpendicular to the total Bergers vector in the primary plane and the effect of impurities upon the formation of constrictions, explain the orientation dependencies in both stoichiometric $L1_2$ and tertiary $L1_2$ alloys.

For the new unpinning mode, we assume, similarly as in the PPV model, that only small sessile segments, i.e. pinning points, are formed. We then develop a new mechanism for thermally assisted unpinning and demonstrate that all the above mentioned features of the anomalous regime can be explained without any additional assumptions. The reason why we propose that pinning points rather than fully developed sessile segments are formed, is that glissile-sessile transformations occur on moving dislocations and as a sessile segment is being created the dislocations bows out around it away from the screw character, preventing its spreading. Unlike in Vitek et al (1991), we consider that the thermally activated unpinning always triggers a major breakaway, i.e. release from many pinning points. The unpinned dislocation then glides freely before being pinned again and the distance travelled is calculated from the condition for steady state in which the average number of pinned segments remains constant. At low temperatures, when the mean separation between the pinning points is large, the release from a single pin is rate controlling. However, as the temperature and the stress increase, major breakaway becomes unlikely after the release from a single pin since re-pinning becomes more probable than further unpinning. The release is then controlled by more difficult, but alternate, reaction paths that involve multi-pin activations. The change in the dominant reaction path is then the reason for the discontinuity in the temperature dependence of the activation volume. (For more details see the enclosed preprints).

Al_3Ti in DO_{22} Form

In the last annual report we described the results of dislocation core simulation in a model DO_{22} structure. It was shown that many possible dislocations exist in the DO_{22} structure, but all have non-planar cores due to the tetragonality of the crystal structure, and are, therefore, sessile. This, in our opinion, is the main reason for the extreme brittleness of this material rather than lack of possible slip systems, as often assumed.

However, the most commonly-observed deformation made in DO_{22} Al_3Ti and Ni_3V is twinning on the {111} planes in the <112> direction. The results of our modelling of dislocation cores under applied stress have provided a mechanism for the nucleation and growth of these twins. This model involves the successive nucleation, due to the applied stress, of pairs of Shockley partial-like dislocations on planes adjacent to the original {111} plane on which the original (nonplanar) dislocation core exists. As the stress is increased, the partials are formed on successive planes, leading to the broadening of the deformation twin. This mechanism is unlike other mechanisms of twinning in that it involves the successive nucleation of dislocation pairs on each plane, a process which is driven by the applied stress and heavily influenced by fault energies (for more details see the enclosed preprint).

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