

CRYSTAL STRUCTURE CHANGES IN Ni_3Al AND ITS ANOMALOUS TEMPERATURE DEPENDENCE OF STRENGTH

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ABSTRACT

The existence of a structural transformation in Ni_3Al alloys established earlier through X-ray diffraction, dilatometry and TEM investigations are summarised. The results obtained are discussed through a model proposed. The L1_2 structure appears to transform to another L1_2 or to a DO_{22} structure during heating. Such a transformation starts at around 700°C and seems to complete around 1100°C . In the temperature range $700\text{--}1100^\circ\text{C}$ both phases coexist. This causes a tetragonal distortion of the L1_2 lattice giving rise to a tweed morphology in TEM observations. The flow stress studies indicate that the anomalous strengthening behaviour is not only due to the dislocation kind of mechanism as proposed in the literature but also due to the structural changes noticed in this work.

1. INTRODUCTION

Ni_3Al , which has been considered as one of the strongly ordered alloys of L1_2 type is generally believed to be stable up to its melting point. However, the literature survey indicates significant anomalies in the long range order parameter determinations of these alloys. For instance, Aoki and Izumi [1] could get a near theoretical value for S (e.g., 0.98 for stoichiometric Ni_3Al) in many alloys on both sides of stoichiometry. In contrast, Masahashi et al. [2] observed that perfect ordering could not be achieved even with stoichiometric alloys. Some discrepancies have also been noticed in the lattice constant of alloys in the Ni_3Al homogeneity region which have not been satisfactorily explained or accounted for [3]. Similar variations were noticed with regard to thermal expansion coefficients and specific heat data [4]. All these facts raise doubts concerning the current assumptions about the stability of the L1_2 structure in Ni_3Al alloys. However, recently, Ramesh et al. provided the evidences for the existence of a structural transformation in Ni_3Al alloys through studies employing X-ray diffraction (XRD) [5], dilatometry [4], transmission electron microscopy (TEM) [6] etc. These results call for further discussion regarding the nature of the structural transitions involved.

Further, Ni_3Al has been a topic of considerable engineering interest in recent years due to its anomalous positive temperature dependence of strength. In literature, this has been largely explained on the basis of a thermally activated cross slip from the $\{111\}$ glide planes to the $\{100\}$ sessile planes and the driving force for such a cross slip has been postulated to be a lower anti-phase boundary (APB) energy on $\{100\}$ planes as compared to $\{111\}$ planes [7,8]. However, continuing work on Ni_3Al has shown discrepancies with regard to the driving force for the cross slip. One such discrepant remark was made by Noguchi et al. [3] who on the basis of comparing the APB energy and/or stacking fault (SF) energy on several L1_2 ordered alloys have suggested that the lower phase stability of the L1_2 phase with respect to the DO_{22} phase is the origin of the anomalous strengthening for an L1_2 alloy. As Ramesh et al. have shown that the L1_2 structure in Ni_3Al alloys is not stable above 600°C , the same temperature region around which incidentally the peak in yield stress has been reported so far, it is possible that such structural instability might have some influence on the strengthening behaviour of these alloys. In the present paper, the possible structural changes are discussed based on the evidences presented in the references cited above [4,5,6] and the possibilities of such structural transitions influencing the characteristic anomalous strengthening behaviour of these alloys are verified through flow stress measurements.

2. EXPERIMENTAL PROCEDURE

Stoichiometric and off-stoichiometric compositions of Ni₃Al alloys with and without boron addition (0.1 wt.%) were investigated. Homogeneous alloys were produced by induction melting in argon atmosphere and by casting into a split type copper mould. The structural aspects have been dealt through (i) room temperature X-ray diffraction studies pertaining to long range order parameter, analyses of the diffraction intensity profiles and lattice parameter measurements and (ii) High temperature X-ray diffraction and TEM investigations, the experimental details of which are described elsewhere [5,6]. Dilatometry and calorimetry (DSC) experiments were carried out as supportive tests to the structural studies [4].

The flow stress measurements were conducted at different temperatures using a thermo-mechanical simulator (Gleeble 1500 manufactured by Duffers Scientific Inc.) for boron doped nickel rich off-stoichiometric and stoichiometric compositions. In the case of boron doped off-stoichiometric composition the flow stress over a range of temperatures from ambient to 800°C was obtained after subjecting the alloy to three homogenisation treatments namely 1000°C-2 days, 600°C-3 days and 600°C-40 days. Cylindrical specimens (6mm dia & 9mm length) were spot welded with a Pt-Pt 10%Rh thermocouple and were heated by means of a resistance heating. The specimens were heated at a faster rate of 100°C per second and were held for five minutes at the test temperatures to obtain the steady state before the actual flow stress test was performed. The fluctuation in temperature was within $\pm 1^\circ\text{C}$ during the test duration. The tests were performed under a vacuum of 4×10^{-5} torr and at a strain rate of $2 \times 10^{-4} \text{ sec}^{-1}$.

3. RESULTS

3.1 Structural Analysis

3.1.1 Room temperature X-ray diffraction

Long range order parameter & intensity profile studies

The long order parameter studies on the filings of Ni₃Al alloys reveal [5] the order of the L1₂ structure to be near the theoretical value at a temperature of 600°C (for a 2h anneal). As the temperature exceeds 600°C, the order parameter decreases and the L1₂ structure becomes unstable relative to one or more other structures. The intensity profiles reveal [5] a marked broadening of all the lines for the filings annealed at temperatures above 600°C. Also, with increasing temperatures a splitting of both the fundamental and the superlattice reflections is noticed. These results gave the first indication that the L1₂ structure in Ni₃Al alloys is not stable at all temperatures as is widely believed to be the case so far.

3.1.2 High temperature X-ray diffraction

High temperature X-ray diffraction experiments performed for the boron doped off-stoichiometric Ni₃Al over the temperature range from ambient to 1200°C apparently indicate the existence of three distinct regions. A few diffractograms obtained during cooling and heating sequences of the sample are shown in Fig.1 for the fundamental (200) reflection. It can be seen in Fig.1 that at 1200°C, there is no irregularity in the shape of the profile. The order parameter estimated [6] on the basis of an L1₂ structure for this temperature using (100)/(200) pairs of reflections gave a value of 0.9412 which is fairly close to its theoretical value (0.95) determined for this composition. As the temperature is decreased below 1100°C an additional peak appears at the right shoulder (higher 2θ) of the first. With a further decrease in temperature, the intensity of the new peak gradually increases at the expense of the original peak and at 800°C the intensities become almost equal. The intensity of the new peak becomes stronger and at temperatures below 600°C, the original peak disappears while the new peak continues to become stronger down to room temperature. The changes occurring during cooling are reversed during heating (right side of Fig.1). The lattice parameters estimated [6] on the basis of an L1₂ structure for the split reflection seen at 800°C (during cooling) showed the lattice parameter of the high temperature phase to be higher by 0.0234 Å as compared with the low temperature phase. A similar difference was noticed in lattice parameter during the heating cycle when calculated for the well resolved split peaks seen at 1000°C. In the following discussions for the sake of simplicity and convenience the low and high temperature phases are designated as L1₂(L) and L1₂(H) respectively.

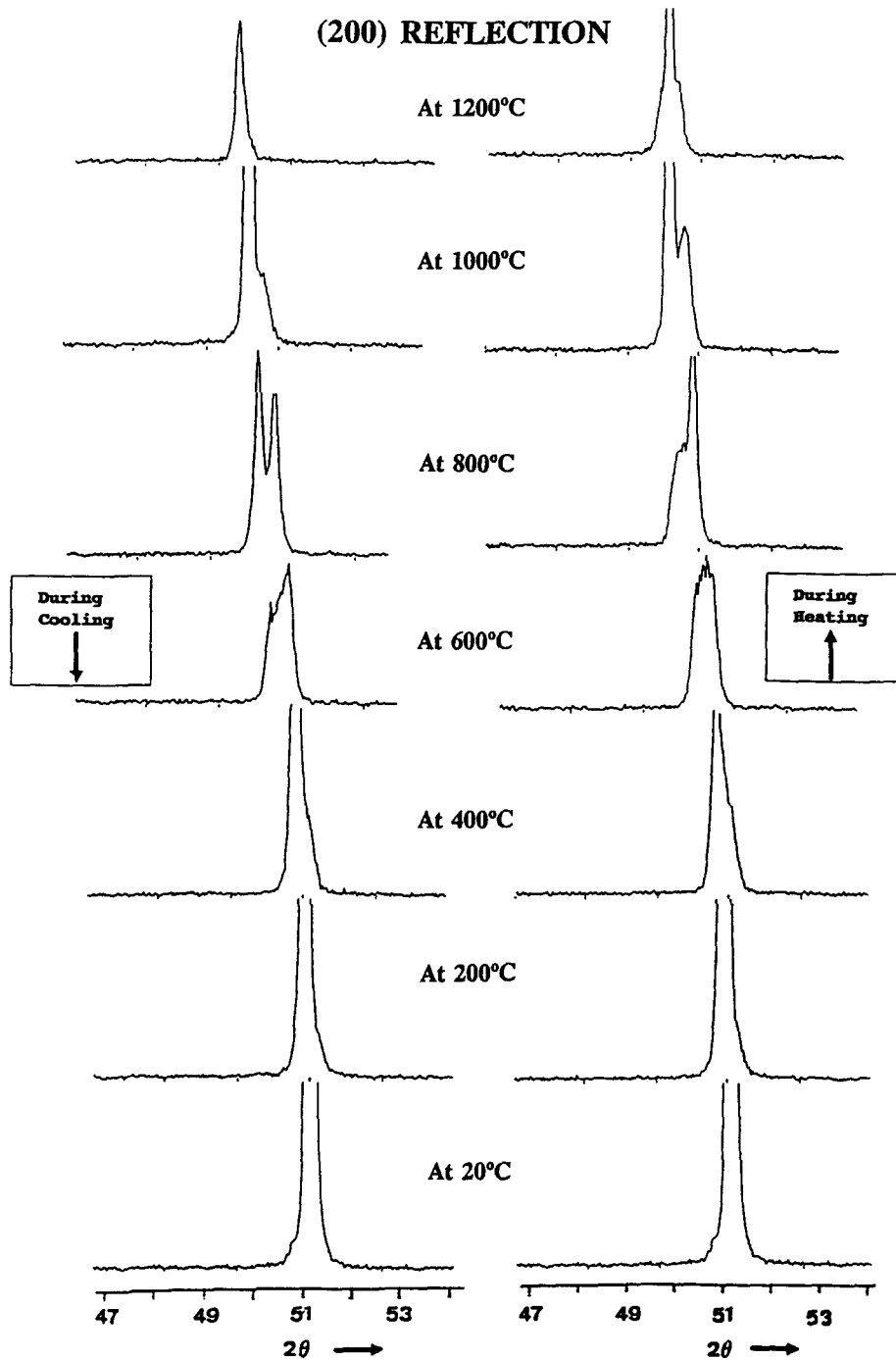


Fig.1 XRD profiles of (200) reflection obtained over the range of temperatures for the boron doped off-stoichiometric Ni₃Al during thermal cycling.

From these results it can be said that there exist three distinct transformation regions namely $< 700^\circ\text{C}$, $700\text{--}1100^\circ\text{C}$ and $> 1100^\circ\text{C}$. It appears that the structure is of $L1_2$ type in the $< 700^\circ\text{C}$ and $> 1100^\circ\text{C}$ regions with the lattice parameter of the latter being larger than that of the former. In the intermediate temperature range the structure(s) appears to be different i.e., a transition from the low temperature $L1_2$ to high temperature $L1_2$ or reverse is more likely.

3.1.3 TEM investigations

TEM investigations were carried out on samples quenched from 1200°C, 1000°C & 600°C corresponding to the earlier mentioned three regions. The microstructures of the samples quenched from 600°C and 1200°C are rather featureless [6] except for the presence of superdislocations while those quenched from 1000°C reveal (Fig.2) the features of a strain (tweed like) contrast along $\langle 110 \rangle$ in a foil oriented in [001]. There is also further evidence from the selected area diffraction patterns that a weak diffuse scattering occurs along $\langle 110 \rangle$ directions for the 1000°C quenched samples while no such diffuse intensity exists for 600°C & 1200°C quenched ones [6].

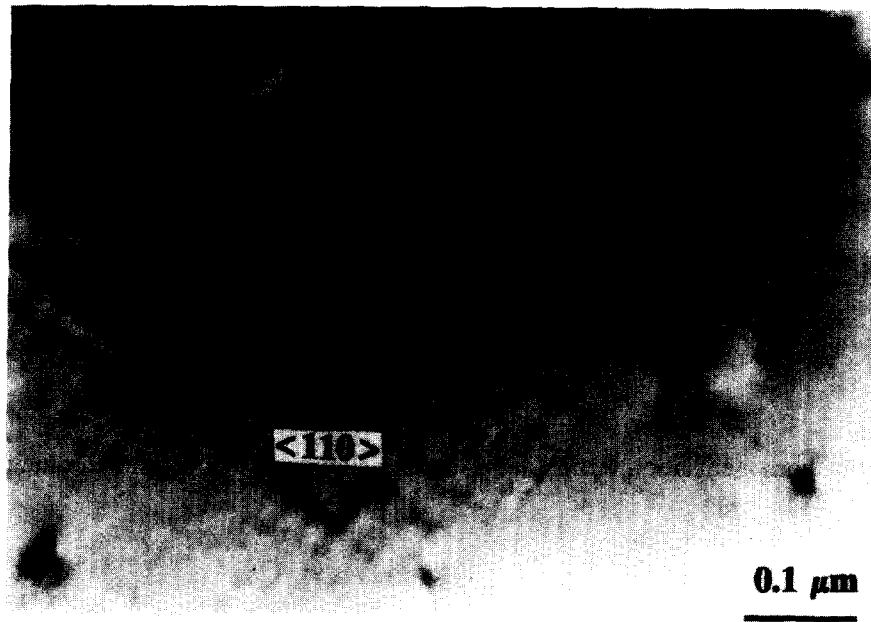


Fig.2. TEM bright field micrograph of boron doped nickel rich Ni_3Al quenched from 1000°C, illustrating the striated contrast along $\langle 110 \rangle$ in [001] direction.

3.1.4. Dilatometry and Differential Scanning Calorimetry

The results of the dilatometry and calorimetry experiments provided additional evidence to that obtained with XRD for a phase transformation in Ni_3Al alloys [4]. Dilatometry isothermal experiments indicated a volume decrease of about 0.45% for the transition from the high temperature structure to $\text{L1}_2(\text{L})$. This is in line with the high temperature XRD results as they indicate a smaller value of lattice parameter for the structure which is stable at room temperature than for that at elevated temperatures. Further, the transition temperatures obtained by the dilatometry and DSC for the transition from $\text{L1}_2 \rightarrow$ intermediate structure were around 650°C and 750°C for off-stoichiometric and stoichiometric Ni_3Al respectively which indicates the composition dependence of the transition temperatures.

3.2 Flow stress results

The flow stress behaviour obtained for the off-stoichiometric boron doped alloy subjected to three different homogenisation treatments is shown in Fig.3. As can be seen from the figure, the alloys homogenised at 1000°C exhibits a higher anomalous strengthening behaviour with temperature as compared to the alloys homogenised at 600°C. Among the two different holding times at 600°C, the alloys homogenised for 3 days exhibits a higher strengthening behaviour as compared to the one homogenised for 40 days. This difference in the anomalous strengthening behaviour for the same alloy subjected to different homogenisation treatments can perhaps be attributed to the structural changes noticed.

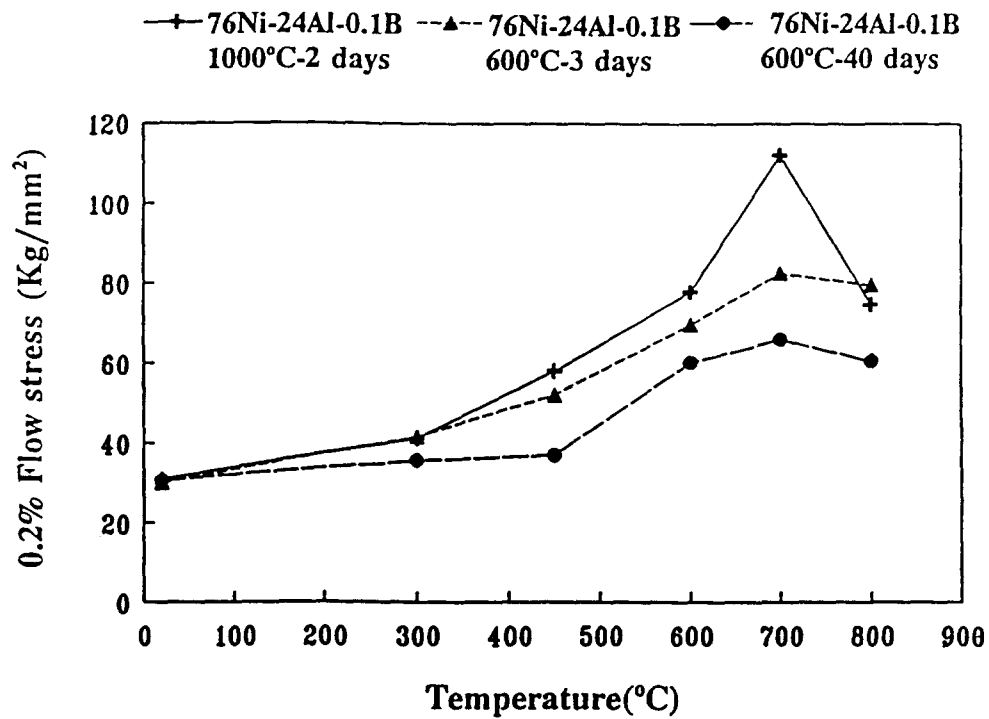


Fig.3. Effect of homogenisation temperatures on the flow stress of off-stoichiometric Ni₃Al.

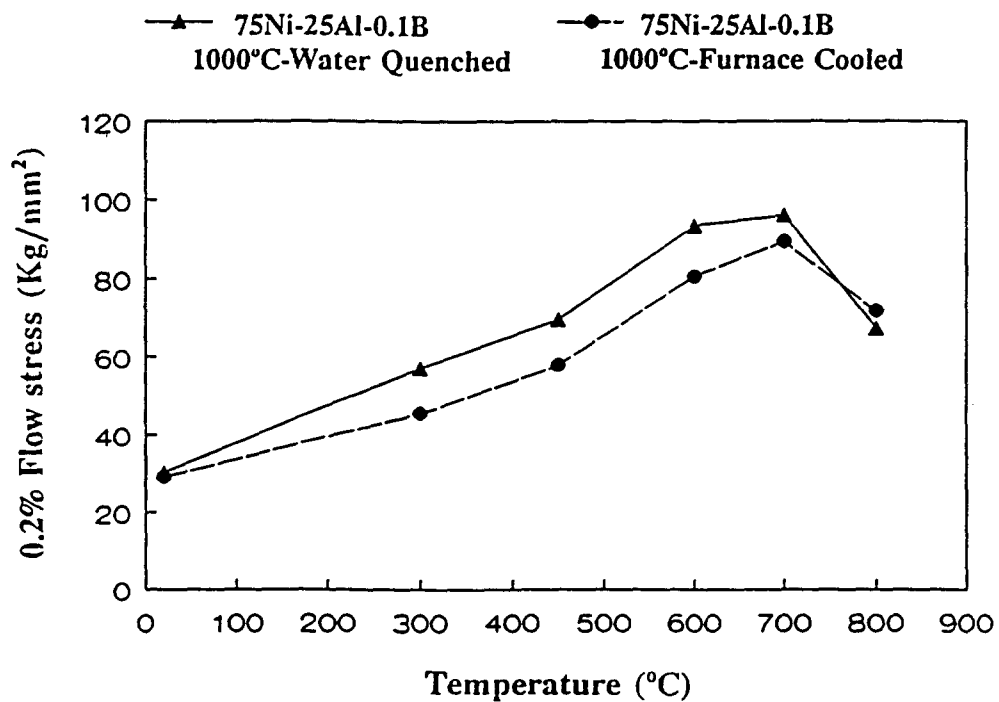


Fig.4. Flow stress as a function of temperature for boron doped stoichiometric Ni₃Al in two different initial conditions.

The alloy (homogenised at 600°C) containing predominantly a low temperature $L1_2$ structure exhibits a lower anomalous strengthening behaviour as compared to that (homogenised at 1000°C) containing the high temperature structure. Between the two different holding times at 600°C, the alloy homogenised at 40 days is expected to contain larger volume fraction of low temperature $L1_2$ phase and hence can have a lower strength as compared to the 3 days homogenised sample which can contain still some of the high temperature phase along with the low temperature $L1_2$. As the measured grain sizes were about 200 & 300 μm for the 600°C homogenised (3 days & 40 days respectively) samples and about 450 μm for the 1000°C homogenised sample, it is clear that they cannot contribute to the differences in the anomalous mechanical behaviour. From the above flow stress results, it can be said that more stable the $L1_2(L)$ structure is, the less can be the anomalous strengthening behaviour.

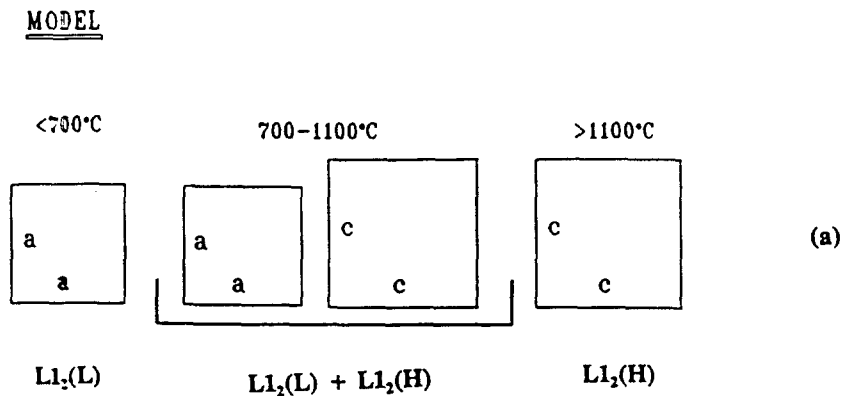
The flow stress behaviour for the boron doped stoichiometric composition in two different initial conditions namely furnace cooled & water quenched after holding at 1000°C for 2 days is shown in Fig.4. Based on the earlier result of Fig.3, the observations made in Fig.4 is in line with the expected trend that the quenched sample which could contain more of the high temperature structure should show higher strength as compared to the furnace cooled one.

4. DISCUSSION

4.1 Structural Analysis

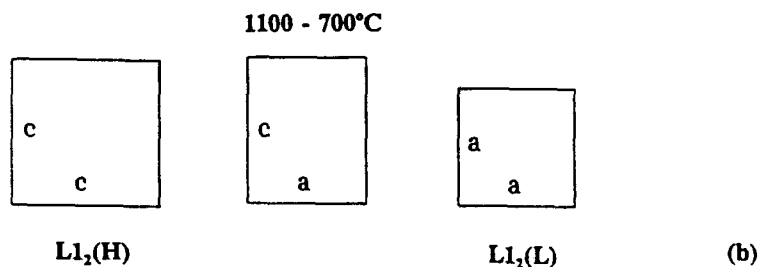
From the high temperature X-ray diffraction measurements it appears that for temperatures below 700°C and above 1100°C an ordered cubic structure of $L1_2$ type exist. The ordered nature of these structures was confirmed through the presence of superlattice reflections [6]. TEM microstructural investigations clearly indicated the presence of superdislocations in these temperature ranges, which further confirms the ordered nature of the structures. However, the cubic structures mentioned in the two regions are not identical as the lattice parameter calculated for the high temperature structure [$L1_2(H)$] was found to be larger by 0.0234Å as compared to the low temperature cubic structure [$L1_2(L)$].

A transition involving the same kind of ordering has not been reported and is difficult to understand from a physical point of view. In this regard, it is pertinent to remark that a close



MECHANISM

During Cooling



During Heating

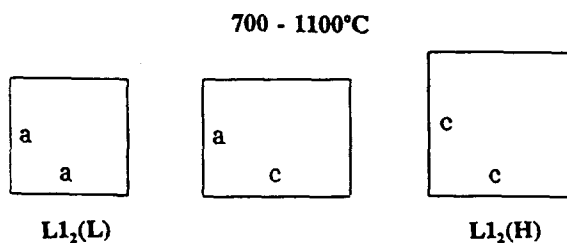
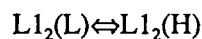


Fig.5 (a) Model describing the proposed structural changes that could possibly exist in Ni_3Al alloys and (b) structural changes from the mechanistic point of view.

structural similarity exists between the $L1_2$ and DO_{22} ordered structures and for that reason the possibility of DO_{22} being the high temperature is considered at a later stage. For the sake of discussions the high temperature phase is first assumed to be an $L1_2$ type ordered structure. Based on the results available, the possible changes that occur in the intermediate region (700–1100°C) are discussed through a model proposed below.

Model

It is postulated above that on either side of the intermediate region (700–1100°C) the structure is ordered cubic ($L1_2$), with the cell parameter being smaller for the cubic structure that exists below 700°C [$L1_2(L)$] than for the one existing above 1100°C [$L1_2(H)$]. Although the in between temperature range is extended, the high temperature X-ray diffraction did not give any indication for the existence of a principal different lattice between 700 and 1100°C. Therefore, in the intermediate region only the transformation takes place and one can expect a mixture of these two different cubic structures:



This structural change is shown in Fig.5a.

The existence of two cubic structures [$(L1_2(L)+L1_2(H))$] in the intermediate region in varying amounts depends on the temperature (Fig.5a). By proposing that the amount of two cubic structures varies with temperature, it is meant that one cubic structure is transforming continuously to the other in the intermediate transition region. However, from a mechanistic point of view (not thermodynamically) such a transition might occur through a metastable state of a localised tetragonal distortion of the lattice. This mechanistic view of the model is shown in Fig.5b. It predicts the $L1_2(L) \leftrightarrow L1_2(H)$ transition to occur through a tetragonal distortion of the parent lattice in the intermediate region.

The validity of the proposed model is discussed in relation to the results obtained with X-ray diffraction and TEM investigations as follows. The (200) intensity profiles (Fig.1) indicate a single peak at 1200°C corresponding to $L1_2(H)$, and as the sample is cooled through the intermediate temperature range (1100–700°C) the emergence of an additional peak followed by its increase in intensity at the expense of the original peak is observed. This gives the idea that $L1_2(H)$ is continuously transforming to $L1_2(L)$. Below 600, the original peak disappears and the new peak becomes stronger until ambient temperature is reached. This new peak corresponds to $L1_2(L)$. On the other hand, when the sample is heated through the same range, the above process can be seen to reverse with the $L1_2(L)$ peak now becoming weaker at the expense of $L1_2(H)$ beyond 600°C. From this one can confirm the existence of $L1_2(L) \leftrightarrow L1_2(H)$ and also the reversibility of the transition.

The TEM microstructures which reveal a featureless microstructure except for the presence of superdislocations for 600°C and 1200°C [6] quenched samples indicate the existence of a single phase ordered structure at the respective temperatures. For the samples quenched from the intermediate temperature region (Fig.2), the features reveal mottled strain contrast or tweed patterns at many locations in the matrix. It has been shown that the tweed contrast arises from regular patterns of (invariably) tetragonal strain centres in the parent cubic lattice [9]. Further Ni_3Al (γ') precipitates which are the strengthening constituents in Inconel 718 superalloy are believed to undergo a $\gamma \rightarrow \gamma'_{\text{bct}}$ transformation based on its tweed morphology when aged around 700°C [10,11]. However, based on our high temperature X-ray diffraction study, the existence of a stable tetragonal phase for Ni_3Al can almost certainly be excluded. The probable cause for the observed tweed formation in the intermediate temperature region of the present study can be attributed to the localised strain induced by a periodic modulation.

The possible existence of a modulated structure in the intermediate region is supported by the room temperature X-ray intensity profiles obtained from the filed samples which were water quenched from 1000°C, the experimental details of which are provided in the reference [5]. The intensity profiles indicate the emergence of satellites around the (111) reflection (Fig.6). Apparently six satellites, three on the low angle side and three on the higher angle side of (111) reflection are noticed which are symmetrical on either side. Higher order reflections did not show any satellites which may be related to their weak intensities. Also the in-situ high temperature X-ray diffraction did not reveal such satellites at 1000°C and this is attributed to the thermal scattering and grain size effects. The available data were evaluated by means of the Daniel-Lipson relation [12] and the L values (distance expressed in number of unit cells in the [100] direction) estimated based on the (111)

reflection for both 1000°C-24 h water quenched (Fig.6a) and 1000°C-1 h quenched (Fig.6d) filings were found to be the same ($L = 62, 70, 74$ for the three orders of satellites noticed in both cases). These values are in good agreement with those obtained with TEM wherein the modulations seen in the micrographs when directly measured on enlarged prints yielded L values in the range 60–65. We believe that these periodic modulations involve modulations in lattice parameter.

Summarising, it can be said that during transformation a cell with a different lattice constant is being formed, which consequently creates mismatch stresses. These are so high that it will not be possible for the two structures to coexist as such. In order to accommodate these stresses the lattice changes gradually from the low to the high temperature structure. This creates a modulation with a periodicity comprising 60–70 unit cells. Because of this periodicity satellites are seen. This phenomenon is very much alike what one can observe with spinodal decomposition. It should be pointed out, however, that in case of spinodal decomposition these modulated structures are due to compositional variations which give rise to strain (lattice constant) modulations, as generally the atomic size of the species are not equal. In our case there is no compositional modulation, but only a lattice and thus a strain modulation is present.

So far in the discussion we have referred the high temperature phase as an ordered cubic structure of $L1_2$ type. As mentioned earlier the occurrence of a transition involving the same kind of ordering as $L1_2(L) \leftrightarrow L1_2(H)$ discussed above is difficult to understand from the physical point of view. On the other hand, a transition from one ordered structure to another ordered structure is more likely and a number of such cases have been reported (e.g., $B_2 \leftrightarrow DO_{22}$ transition in FeAl system [13]). A good structural similarity exists between $L1_2$ and DO_{22} ordered structures. The tetragonal DO_{22} unit cell is composed of two unit cells of $L1_2$ shifted with respect to each other on the (001) boundary plane by an amount $1/2 a_1 + 1/2 a_2$, (i.e. by $1/2 a[110]$). It is often referred to as "long period" superlattice (one dimensional) of the $L1_2$ structure with $M=1$ [14]. In the literature the probability of a transition occurring between $L1_2$ and DO_{22} has been reported only as a likelihood and no experimental confirmation to such a transition exists. In this regard, Noguchi et al. [3] have predicted that an increasing alloying addition can cause an $L1_2$ phase in Ni_3Al alloys to become unstable with respect to DO_{22} . If this is so, it may be quite possible that even the temperature difference can cause the $L1_2$ phase to become unstable with respect to DO_{22} . In such a case, the observed high temperature phase designated as $L1_2(H)$ can

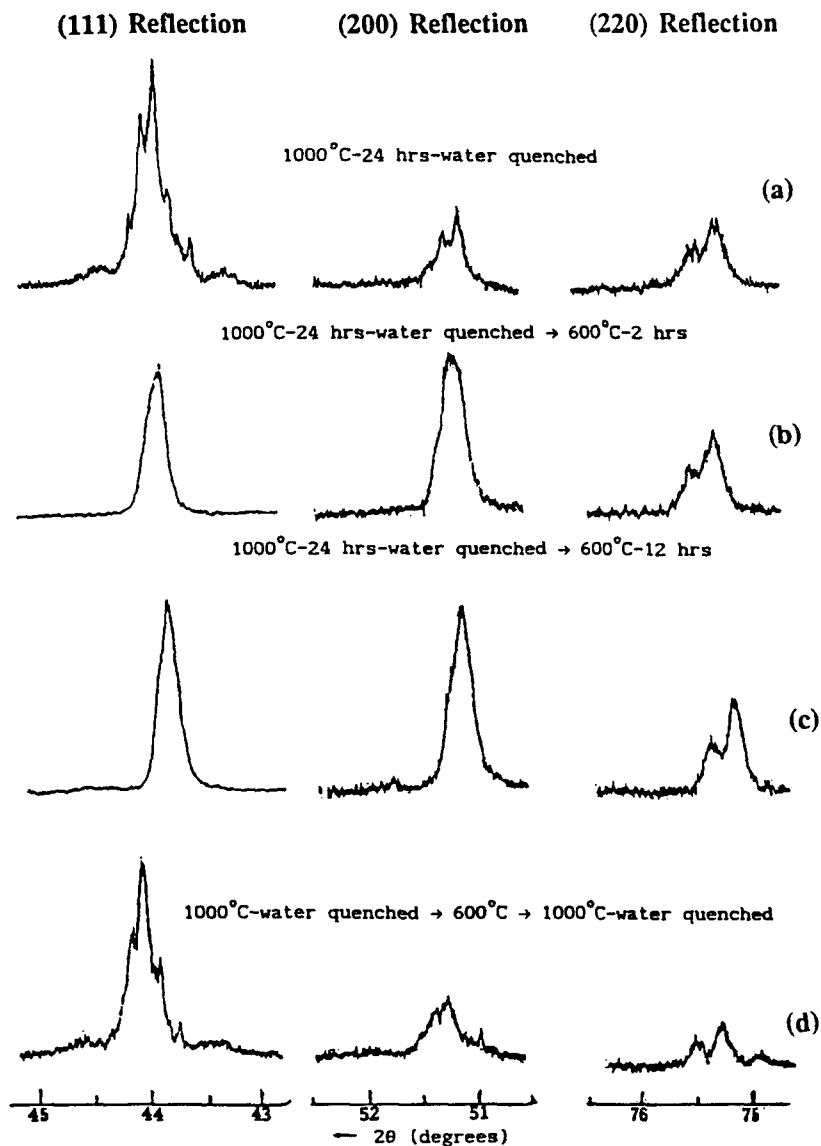


Fig.6. XRD intensity profiles of boron doped nickel rich Ni_3Al providing the evidence for the existence of a modulated structure in the intermediate temperature region.

well be a DO_{22} ordered structure. The prerequisite is that it has an a axis little larger than $L1_2(L)$ and equal to $1/2 c$, in order to fit the XRD results. More experimental studies involving TEM, single crystals and rigorous thermodynamic analysis would be necessary if unequivocal confirmation to the presence of DO_{22} structure is to be provided.

4.2 Anomalous positive temperature dependence of strength

As stated earlier, the theoretical attempts to explain the anomalous strengthening behaviour through phase stability concepts were made by Noguchi et al. [3] and Wee et al [15]. Their argument is, if the cross slip process is favoured due to the low APB energy (at elevated temperatures) on $\{100\}$ planes causing the anomalous strengthening in this crystal structure as has been widely reported, then this does not satisfactorily explain as to why many other $L1_2$ ordered alloys do not show similar anomalous strengthening behaviour. For example, $L1_2$ ordered alloys like Ni_3Fe , Pt_3Al , Ir_3Cr show the usual negative temperature dependence of strength [15]. On the basis of these findings Wee et al. and later on Noguchi et al. have suggested that the high APB energy on $\{111\}$ planes is not a prerequisite for the anomalous behaviour and that the lower phase stability of the $L1_2$ phase with respect to the DO_{22} phase is indeed the origin of the anomalous strengthening in an $L1_2$ ordered alloy. Further, recent studies indicate that the measured APB energies for the (111) and (100) planes of the $L1_2$ structure are similar and this suggest that the differences in APB energy is not a significant driving force for the cross slip [16]. These new results further stimulate to consider the arguments of Wee & Noguchi et al. more seriously that the relative phase stability of $L1_2$ structure with respect to DO_{22} or some other structure could be the origin for the anomalous strengthening behaviour.

The present study has provided the first experimental evidence for the phase instability of the $L1_2$ structure. In the following discussions an attempt to relate these structural changes to the anomalous strengthening behaviour is made.

The analysis of the results of Fig.3 indicates that more is the $L1_2(L)$ structure present, the lower is the anomalous strengthening behaviour. In other words, more is the high temperature phase present, the higher is the anomalous strength. This is confirmed from the results of Fig.4 wherein the water quenched (from $1000^\circ C$) sample exhibited higher strength as compared to the furnace cooled one as the former sample is expected to contain more of the intermediate phase compared to the latter. An explanation of this nature can lead to a question that if the presence of the high temperature phase can cause an increase in strength then why the strength should decrease beyond $700^\circ C$ where the high temperature phase is supposed to be more predominant. This can perhaps be answered by considering the flow stress curve obtained for a $600^\circ C$ -40days homogenised sample (Fig.3) and the $1000^\circ C$ water quenched specimen (Fig.4) which can reasonably be treated as representing the flow stress curves for an $L1_2$ and the high temperature structure respectively. In both cases, the flow strength increases with increasing temperature and decreases beyond $700^\circ C$ though in the former case the rate of increase is much lower as compared to that in the latter. The fact that the flow stress decreases beyond $700^\circ C$ in both cases draws us to a conclusion that the 'thermal effect' on flow stress dominates over the 'structural transition hardening effect'. On the other hand, below the temperature of peak strength the presence of the intermediate phase influences the strength anomaly rather significantly. Our argument that the high temperature phase enhances the strength is in good agreement with the results of Cheng et al. [17] who found similar increase in strength for the proton irradiated Ni_3Al possessing a distorted tetragonal structure together with the equilibrium $L1_2$ compared to an unirradiated Ni_3Al possessing only the $L1_2$ structure. The $\gamma' \rightarrow \gamma_{bct}$ transformation seen in Inconel 718 also retains the high degree of hardening [11]. These examples support our argument that the high temperature phase enhances the strength.

The reasons for such an increase in the anomalous strength with the increase of the content of high temperature phase may be attributed to the presence of internal stresses. It has been explained earlier that the intermediate temperature region possess one of a distorted or a strained lattice resulting due its accommodation of the product phase through an elastic deformation of the parent lattice. Elastic deformation of the lattice produces internal stresses or coherency stresses. Tweed microstructures provided the evidence for the existence of a distortion in the lattice. So the material (quenched from $1000^\circ C$) possessing substantially the high temperature phase is expected to have more of these internal stresses. This can enhance the drag stress for the dislocation motion resulting in an increased anomalous strengthening behaviour. At test temperatures above $700^\circ C$, the temperature effect softens the lattice (annihilation of dislocations, defects etc.) resulting in the usual decrease of strength with increasing of temperature.

5. CONCLUSIONS

1. The $L1_2$ structure in Ni_3Al alloys is not stable at all temperatures as is widely believed to be the case so far.
2. There exists three different phase regions namely $<700^\circ C$, $700-1100^\circ C$ and $>1100^\circ C$ in Ni_3Al alloys. The structures are $L1_2(L)$ and $L1_2(H)$ or DO_{22} in the $<700^\circ C$ and $>1100^\circ C$ temperature regions respectively. In the intermediate region, there is a coexistence of these two structures.
3. The transition involves a tetragonal distortion of the parent $L1_2$ lattice and modulations in lattice parameter.
4. The noticed structural changes influence the anomalous strengthening behaviour of these alloys. That is, the larger is the presence of the high temperature phase, the higher is the strength at any temperature up to $700^\circ C$.
5. The anomalous strengthening behaviour is not only due to the dislocation kind of mechanism proposed in the literature but also due to the above discussed structural changes.

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