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## Abstract

The mechanical properties and dislocation microstructure of single crystals with a range of compositions within the Fe<sub>x</sub>-Ni<sub>60-x</sub>-Al<sub>40</sub> pseudobinary system have been investigated, with the purpose of bridging the behavior from FeAl to NiAl. Experiments are focused on the compression testing of  $\langle 001 \rangle$  oriented single crystals with compositions where  $x = 10, 20, 30, 40,$  and  $50$  (in atomic percent). Observations of  $a\langle 111 \rangle$  dislocation morphologies at room temperature and both  $a\langle 111 \rangle$  and non- $a\langle 111 \rangle$  dislocation activity at elevated temperatures are reported and discussed. Measurements of the yield strength, elastic modulus and strain hardening rates are reported, and the variation of strength with composition is correlated with dislocation dissociation and overall dislocation morphology.

## Disciplines

Metallurgy

## Comments

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## Dislocation Processes and Deformation Behavior in <001>-Oriented Fe<sub>x</sub>-Ni<sub>60-x</sub>-Al<sub>40</sub> Single Crystals

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### ABSTRACT

The mechanical properties and dislocation microstructure of single crystals with a range of compositions within the Fe<sub>x</sub>-Ni<sub>60-x</sub>-Al<sub>40</sub> pseudobinary system have been investigated, with the purpose of bridging the behavior from FeAl to NiAl. Experiments are focused on the compression testing of <001> oriented single crystals with compositions where  $x = 10, 20, 30, 40,$  and  $50$  (in atomic percent). Observations of  $a\langle 111 \rangle$  dislocation morphologies at room temperature and both  $a\langle 111 \rangle$  and non- $a\langle 111 \rangle$  dislocation activity at elevated temperatures are reported and discussed. Measurements of the yield strength, elastic modulus and strain hardening rates are reported, and the variation of strength with composition is correlated with dislocation dissociation and overall dislocation morphology.

### 1. INTRODUCTION

Extensive research has been conducted with respect to the binary NiAl and FeAl intermetallic compounds having the B2 crystal structure [1-3]. Their attractive high temperature properties, relatively high yield strengths, and resistance to oxidation and corrosion have piqued interest in these systems. Unfortunately, poor low-temperature ductility, inadequate fracture toughness, and rapid loss of strength at high temperatures have been critical negatives for potential applications. Nevertheless, from a fundamental viewpoint, these alloys represent an ordered intermetallic system in which complex point defect and dislocation behavior play a strong role in governing the deformation mechanisms. NiAl alloys show higher room temperature yield strengths than FeAl, but little-to-no room temperature tensile ductility. Their deformation via  $a[001]$  dislocations provides only three independent slip systems, falling short of the five slips systems required for plastic flow (in polycrystals) by the Von Mises criterion. FeAl deforms with a lower room temperature yield strength, and exhibits significant (although somewhat variable) ductility. The larger ductility has been explained by the fact that active  $a\langle 111 \rangle$  dislocations provide sufficient slip systems to satisfy the Von Mises criterion. There is also evidence that hydrogen embrittlement and weak grain boundaries are also important factors limiting the overall ductility [3].

Of particular interest in this study is the effect of large solute additions on the properties of ternary compositions within the (Ni,Fe)-40Al pseudobinary system, with which we are able to bridge the behavior from NiAl to FeAl binary B2 compounds. In early (Ni,Fe)-Al pseudobinary work by Nagpal and Baker [4], the hardness as a function of composition was measured for alloys containing 45%Al. A dramatic influence of quenching rate for Fe-rich compositions was also observed. Patrick, et al [5] performed a TEM study of dislocation structures in polycrystals over a range of compositions with 40%Al. They determined that a change from  $\langle 111 \rangle$  to  $\langle 100 \rangle$  slip occurs in the composition range between 30Fe-30Ni-40Al and 50Fe-10Ni-40Al. Most recently, Pike [6-7] has determined the site occupancy, point defect concentration, and hardness as a function of composition. Furthering these (Ni,Fe)-40Al pseudobinary studies with the correlation of mechanical properties and dislocation mechanisms should provide significant insight into the parameters that control deformation, and may provide solutions to the critical drawbacks inherent in the FeAl and NiAl systems. The present studies have focused on the behavior of  $a\langle 111 \rangle$  dislocations in hard oriented [001] single crystals as a function

of composition, both at room temperature and elevated temperatures. Motion of  $a\langle 111 \rangle$  dislocations in  $[001]$  oriented crystals is induced because there is no resolved shear stress for the glide of the  $\langle 001 \rangle$  slip vectors dominant in polycrystalline NiAl deformation. As the  $a\langle 111 \rangle$  dislocation is the desired slip vector for ductility, a characterization of its nature versus composition and temperature is crucial to understand and optimize the mechanical properties of the pseudobinary system. A complementary set of polycrystalline samples are also being investigated in order to investigate their corresponding strengths and potential for ductility.

## 2. EXPERIMENTAL

Single crystals in the  $\text{Fe}_x\text{-Ni}_{60-x}\text{Al}_{40}$  composition range were prepared at the Materials Preparation Facility of the AMES Laboratory at Iowa State University using the Bridgman method. Note that all compositions will be in atomic percent. Following homogenization at  $1000^\circ\text{C}$  for 72h,  $[001]$  oriented compression samples were electro-discharge machined in the form of  $3 \times 3 \times 8$  mm parallelepipeds. The side faces were  $\{010\}$  oriented and electropolished to facilitate optical slip trace analysis after mechanical testing. The compositions investigated were 10Fe-50Ni-40Al, 20Fe-40Ni-40Al, 30Fe-30Ni-40Al, 40Fe-20Ni-40Al, and 50Fe-10Ni-40Al. Single crystal mechanical data for Ni-40Al and Fe-40Al from Noebe [8] and Yoshimi [9] are reported for comparison purposes. The actual compositions and impurity levels for the single crystals are given in Table 1.

**Table 1:** Nominal and actual compositions and impurity levels for the single crystals.

Nominal Composition	at%						ppm by weight		
	Ni	Fe	Al	Si	Ti	Hf	C	O	N
10Ni-50Fe-40Al	10.9	50.2	38.9	160	<90	<20	<10	17	3
20Ni-40Fe-40Al	20.6	39.7	39.7	<70	<5	<10	24	72	4
30Ni-30Fe-40Al	31.5	30	38.5				23	10	<1
40Ni-20Fe-40Al	39.7	20.4	39.9	<50	<5	<10	13	34	1
50Ni-10Fe-40Al	49.6	10.6	39.9	70	<20	<5	44	15	3

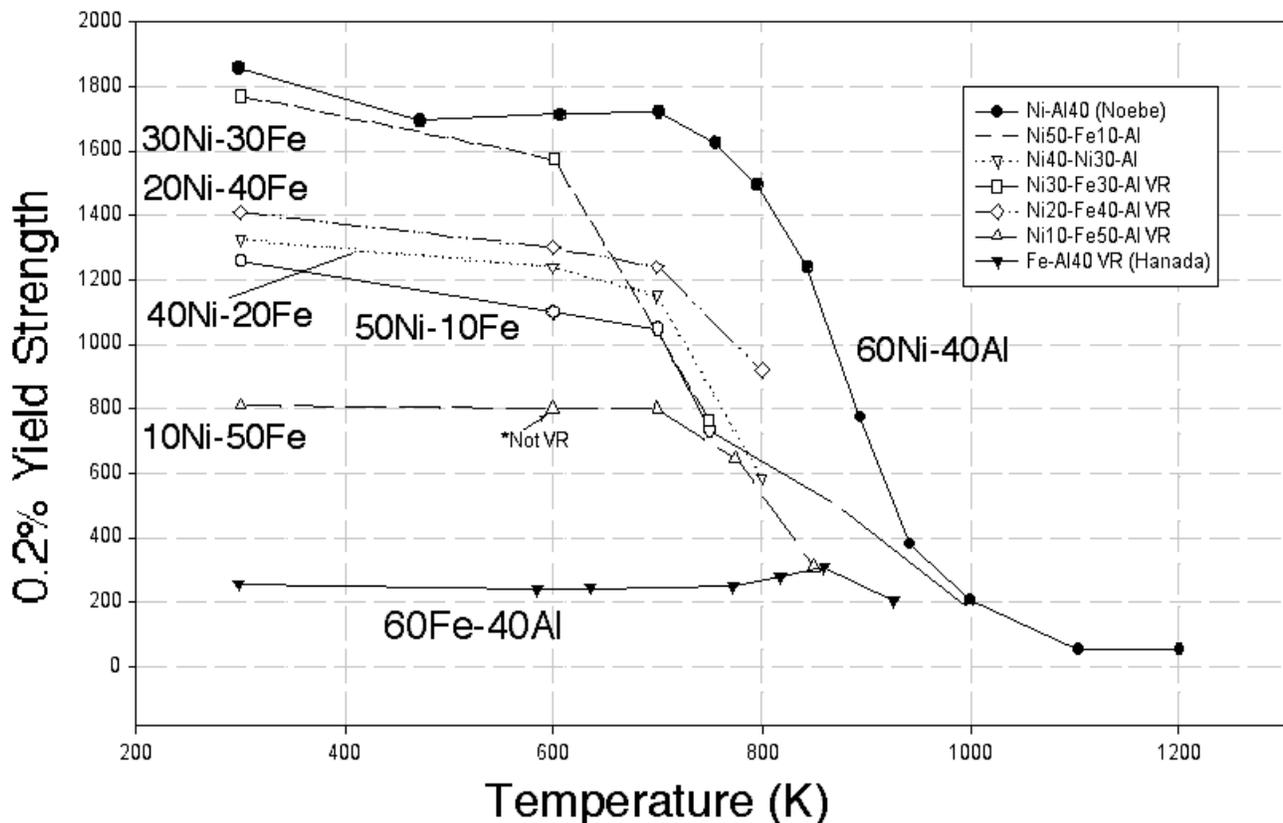
Compression samples of the Fe-rich compositions (ranging from 10-30 % Fe) were sealed in quartz tubes evacuated (to  $10^{-6}$  torr) and backfilled with high purity argon. These samples were subjected to vacancy reducing heat treatments at  $450^\circ\text{C}$  of varying durations (10-40 days), and hardness variations were monitored with microhardness measurements in order to insure that an equilibrium vacancy content had been obtained [10]. These vacancy annealing conditions have been previously established by Schneibel, et al. [11] as an effective treatment. Following suitable vacancy reducing heat treatment, the samples were tested in an ambient environment using an Instron model 1362 equipped with a MAR-M246 compression cage. Displacement was measured with LVDT's mounted on the platens of the cage. The tests were all performed at a constant strain rate of  $10^{-4}/\text{s}$  to a final plastic strain of 1-4%.

Thin foils were prepared for transmission electron microscopy (TEM) with foil normals either in the direction of the compression axis  $[001]$  or normal to an active slip plane. The foils were cut from the compression samples using a low speed saw equipped with a SiC abrasive wheel. Final electrojet polishing was performed at  $-40^\circ\text{C}$ , at 25-30 mA and 4-8 V, on a Tenupol-3 twin-jet electropolishing unit using an electrolyte of 25% nitric acid in methanol for the Fe-rich samples and an electrolyte of 10% perchloric acid in methanol for the Ni-rich samples. Transmission Electron Microscopy (TEM) studies were performed on a Phillips CM200 LaB<sub>6</sub> electron microscope operating at 200kV.

### 3. RESULTS

#### 3.1 Mechanical Behavior

The 0.2% yield strengths for each composition versus testing temperature is shown in Figure 1, along with the aforementioned data from Noebe [8] and Yoshimi [9]. This graph reveals the high yield strength of the 30Fe composition, the high to moderate yield strength of the 20Ni, 40Ni, and 50Ni compositions, and the moderate strength of the 10Ni composition. While the data as a function of temperature is rather sparse, it is clear that the variation of the yield strength with temperature is relatively modest at lower temperatures for all compositions. In addition, all five ternary alloys demonstrated a “knee” in yield strength at elevated temperatures, above which the temperature dependence is significantly larger. The appearance of such a “knee” has previously been correlated with a transition from  $\langle 111 \rangle$  dislocations at lower temperatures to non- $\langle 111 \rangle$  activity at higher temperatures [12-15]. As discussed below, a similar transition appears to be occurring in the present case. The pronounced decrease in yield strength is evident in the approximate temperature window from about 600 to 850K; with the initial transition temperature varying significantly with composition. It is indeed interesting to note that all alloys show the slip transition (associated with the sharp decrease in yield strength) in a narrow temperature window, even though there are significant differences in the dislocation configurations in these alloys at lower temperatures, as will be discussed below. Finally, it is noted that in this temperature regime the ternary compositions are all significantly softer than the binary Ni-40Al compound.



**Figure 1:** Yield strength and composition versus temperature for (Fe,Ni)-Al hard-oriented single crystals tested in compression. “VR” indicates samples subjected to a vacancy reduction heat treatment.

In order to more clearly see the variation of lower temperature strength with composition, Figure 2 presents the single crystal yield strength versus composition at both room temperature and 600K. At

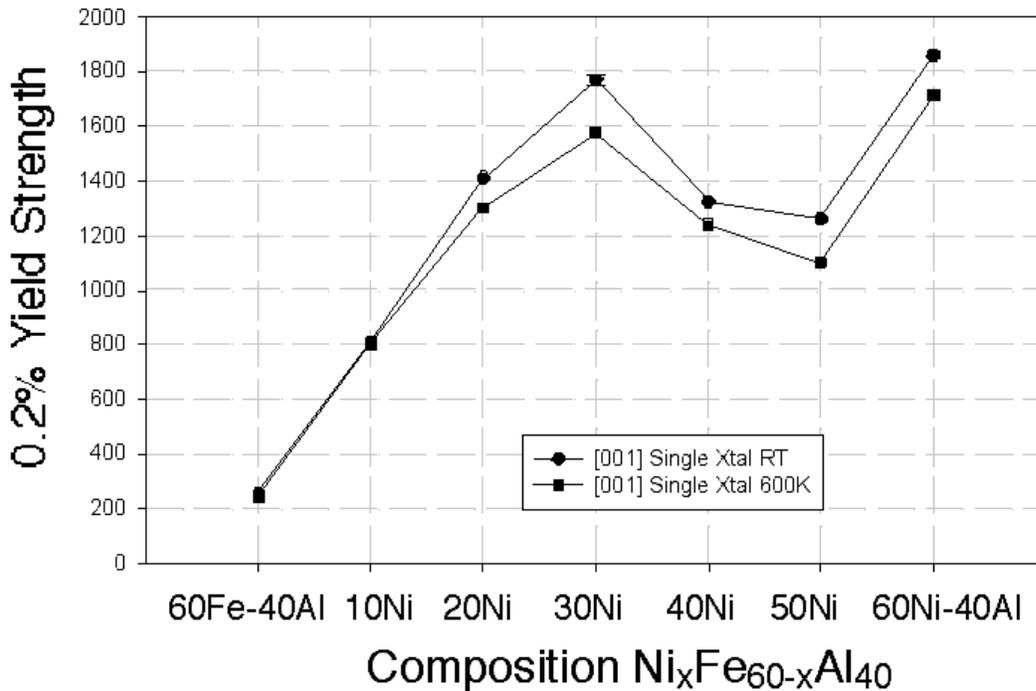


Figure 2: Yield strength versus composition at room temperature and 600K.

both temperatures, there are several striking features to this variation. First is the remarkably strong increase in yield strength for Fe-rich compositions. Second is the peak in yield strength observed at the 30Fe-30Ni-40Al composition. The third prominent feature is the clear solute softening observed for for compositions between Ni-40Al and 10Fe-50Ni-40Al. This variation in yield strength with composition is much more complicated than that observed in isomorphous, binary disordered systems such as Cu-Ni and Au-Ag [16]. In these cases, solute strengthening is always observed upon alloying the pure metals, and the increase in yield strength is classically understood in terms of both size and modulus misfit, based on the Fleisher solid solution model [17]. As discussed below, this model appears incapable of explaining the behavior observed at either extreme of the Fe-Ni-Al pseudobinary system.

Relative measurements of elastic modulus and strain hardening rates were taken from the room temperature compression stress strain curves, but have not been normalized with respect to the inherent moduli of the testing apparatus and compression cage. Elastic constant measurements are presently being conducted, and will be reported elsewhere. Nevertheless, all tests were performed in the same apparatus and thus should provide accurate relative comparisons. As shown in Figure 3a, the elastic modulus increases substantially for the 20Fe and 30Fe compositions relative to the Fe- and Ni-rich compositions. It is interesting to note that this maximum in the modulus values corresponds roughly with the maximum observed in the measured yield strength. The work hardening rates shown in Figure 3b were all measured at a plastic strain of about 2%. It should be noted that strain hardening

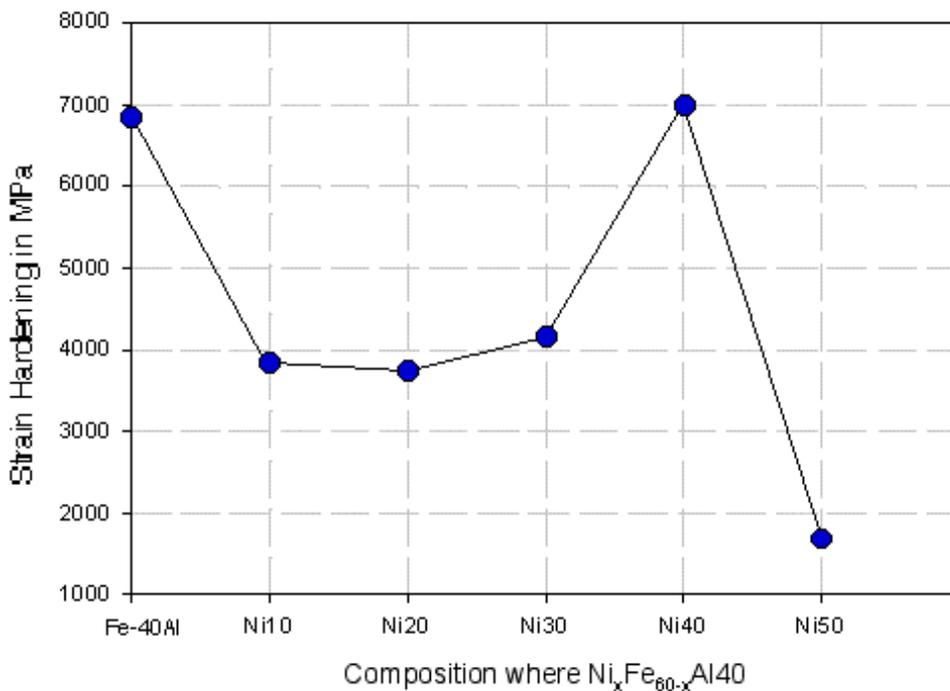
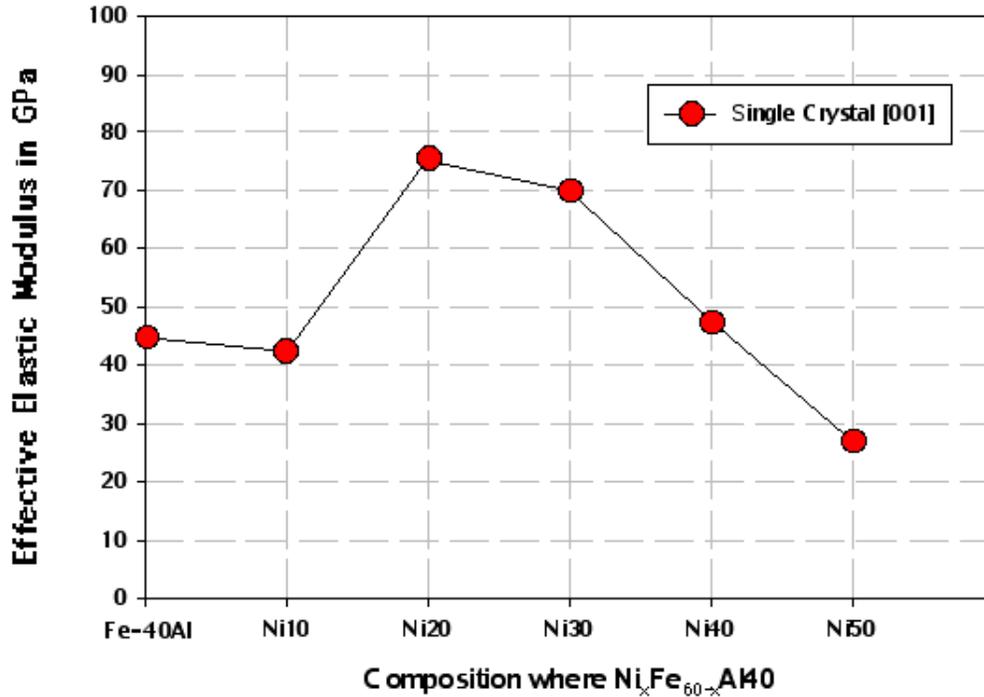


Figure 3: (a) Elastic modulus and (b) strain hardening rate (at ~ 2% plastic strain) measured from compression tests at room temperature.

was nearly linear for most compositions. With the exception of the binary Ni-40Al composition, the strain hardening rates are large for all compositions (ranging from ~ E/6 for Fe-40Al, ~E/10 for 20Fe-40Ni-40Al and ~ E/20 for 40Fe-20Ni-40Al).

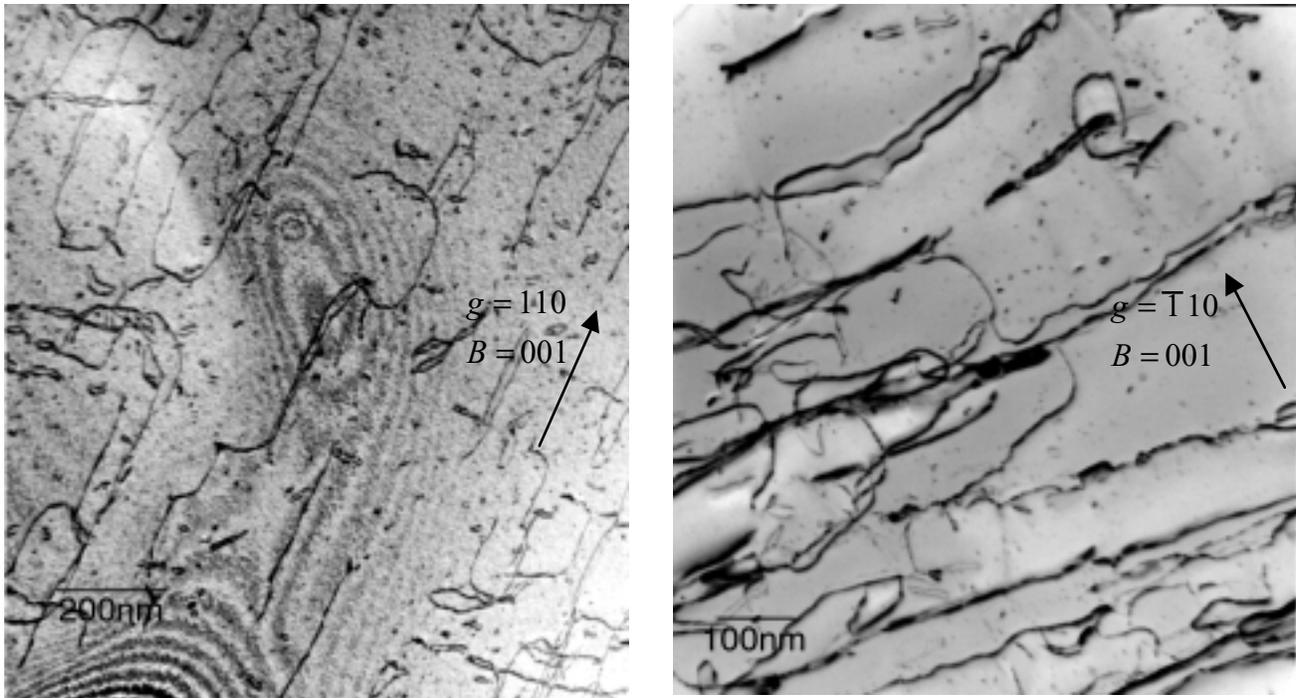


Figure 4: (a) 40Fe-20Ni-40Al single crystal  $\{112\}$  glide plane cut after 2% plastic strain in compression at room temperature.  $a\langle 111 \rangle$  dislocations predominantly in screw orientation. (b) Ni50Fe10Al single crystal  $\{001\}$  compression axis cut after 2% plastic strain in compression at 600K.  $a\langle 111 \rangle$  dislocations predominantly in edge orientation.

### 3.2 Dislocation Observations

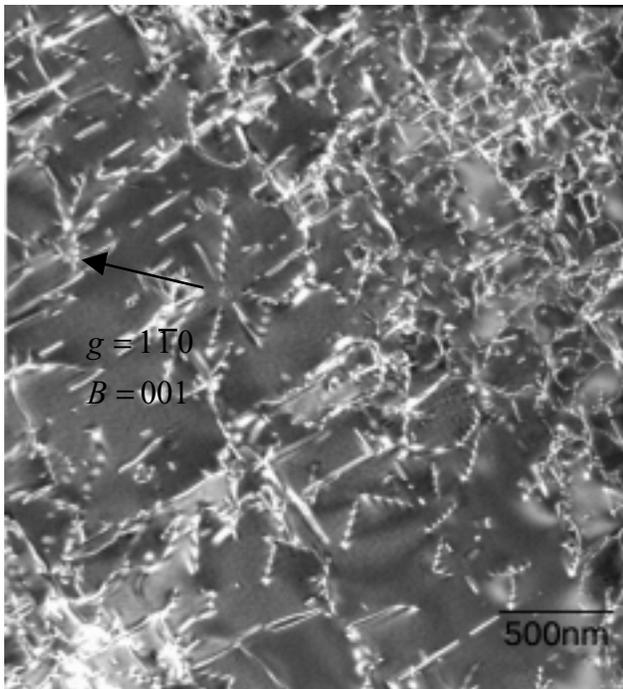
Dislocation analysis was performed to examine morphologies at both room temperature and elevated temperatures. A brief summary of these results is provided here. For all compositions, the deformation microstructures at room temperature and 600K consisted of  $a\langle 111 \rangle$  dislocations, with typically several slip systems operative in a given region, as expected for this high symmetry zone axis in the absence of strong latent hardening effects. There are two noteworthy variations in  $a\langle 111 \rangle$  behavior with composition. First, upon the addition of Ni to Fe-40Al, there is a dramatic decrease in the dissociation distance. Previous work has shown that  $a\langle 111 \rangle$  dislocations in Fe-40Al are dissociated into  $a/2\langle 111 \rangle$  partials [18-19]. TEM observations by Savage [19], and in this work, have shown that the dissociation distance decreases from 3.5nm for Fe-40Al to 1.9nm for 50Fe-10Ni-40Al. No resolvable dissociation of the  $a\langle 111 \rangle$  dislocations has been detected for the 40Fe-20Ni-40Al composition, nor for more Ni-rich compositions. Thus, Ni appears to profoundly increase the APB energy, as expected from simple consideration of the relative ordering energies of NiAl and FeAl.

A second noteworthy change with composition is in the preferred line direction of the  $a\langle 111 \rangle$  dislocations. TEM images for compositions ranging from Fe-40Al to 30Fe-30Ni-40Al reveal  $a\langle 111 \rangle$  dislocations predominantly near screw orientation (see Figure 4a). Somewhere between Ni30 and Ni50 there is a transition from observed  $\langle 111 \rangle$  screws to  $\langle 111 \rangle$  edge line lengths, as exemplified by the image in Figure 4b. This implies that the edge lengths are most mobile in Fe-rich samples, while the screw line direction is more mobile for Ni-rich compositions. This tendency is observed at both room temperature and 600K.

Several transitions are also observed as a function of temperature, the first involving the predominant slip plane. While the Fe-40Al and 50Fe-10Ni-40Al samples deform via slip on  $\{110\}$  planes at both room temperature and 600 K, all compositions with greater Ni content showed a predominance of  $\{112\}$  slip planes at room temperature. However, at 600 and 700K respectively, a transition to slip on  $\{110\}$  type planes is seen for the 40Fe-20Ni-40Al and 10Fe-50Ni-40Al compositions. This transition to  $\{110\}$  slip planes at higher temperatures is believed to be a trend for all Ni-rich alloys.

The second important transition with temperature is that from  $\langle 111 \rangle$  slip at lower temperatures to  $a\langle 100 \rangle$  climb at higher temperatures. This transition, which is common to all the ternary compositions, is exemplified by the dislocation structure shown in Figure 5 for the 10Fe-50Ni-40Al composition deformed at 750K, where several families of  $a\langle 100 \rangle$  dislocations are observed. Note that this sample was deformed at a temperature only about 50K higher than the apparent temperature of the “knee” seen in the yield strength versus temperature plot of Figure 1, and yet no  $a\langle 111 \rangle$  dislocations are present. The common transition to  $a\langle 001 \rangle$  climb should be contrasted with previous work that has proven the operation of  $a\langle 110 \rangle$  glide as the dominant deformation process for binary Ni-rich compositions [20]. Thus, the significantly larger yield strengths for Ni-40Al in the temperature range from 600K to 900K appears to be directly related to differences in the  $a\langle 111 \rangle$  decomposition process, as well as to differences in the deformation mode above the “knee”.

#### 4. DISCUSSION



**Figure 5:** 10Fe-50Ni-40Al single crystal with foil normal parallel to  $[001]$  compression axis after 1.5% plastic strain at 750K. The dominant Burgers vector is  $a\langle 001 \rangle$ .

The very large solute strengthening observed upon the addition of Ni to Fe-40Al, which persists up to 30% Ni additions is clearly associated with the influence of Ni on the critical resolved shear stress for  $a\langle 111 \rangle$  dislocations. Note that these compositions have been carefully annealed in order to minimize the effect of quenched-in vacancies which is known to produce significant strengthening in FeAl [18,21-22]. The strengthening due to Ni does not appear to be consistent with a classical solute interaction for several reasons. Based on ALCHEMI measurements [23], Ni is expected to substitute for Fe on the Fe sublattice. However, there is only a modest size misfit between Ni and Fe based on their Goldschmidt radii [24]. In the Fleischer solute strengthening model [17], the effective misfit due to atomic size and

modulus,  $\epsilon_s$ , is given by:  $\epsilon_s = |\epsilon_G - \beta\epsilon_b|$ , where  $\epsilon_G$  is the modulus misfit,  $\epsilon_b$  is the size misfit and  $\beta$  is a term which accounts for the greater elastic interaction for dislocations with edge character. In this context it is noteworthy that in this composition range  $a\langle 111 \rangle$  screw dislocations are the dominant line direction, indicating their limited mobility. In addition, the overall increase in the elastic modulus in this regime indicates that the shear modulus should also increase. Since the  $\beta\epsilon_b$  term is by convention taken as positive, adding an elastically “stiffer” atom will actually tend to *reduce* the value of  $\epsilon_s$ .

The low mobility of screw dislocations, as evidenced by their long lengths in TEM observations, suggests that a classical Peierls mechanism is operative. It is tempting to further hypothesize that Ni may be altering the core structure of the  $a\langle 111 \rangle$  dislocations, and thus changing the Peierls stress. A second observed trend however argues against this interpretation. For all compositions, the yield strength is only weakly dependent on temperature below the “knee”. The observation of such yield strength “plateaus” which have large stress levels, is difficult to reconcile on the basis of a simple Peierls model for which it is expected that strength will continuously decrease with increasing temperature.

Takeuchi [25] has presented a modification of the Peierls mechanism in the smooth kink regime for dissociated dislocations. He has considered two cases: one in which kink-pair nucleation is correlated on the partial dislocations, and one in which kink pairs are nucleated independently. While in the former case, the yield strength falls continuously with temperature, in the latter case a critical, athermal stress is predicted. This critical stress is associated with the creation of a small additional fault area between a kink-pair on the leading partial dislocation in the case of uncorrelated kink-pair formation. For the present case of an  $a\langle 111 \rangle$  dislocation dissociated into  $a/2\langle 111 \rangle$  superpartials with an APB in between, this critical stress is given by:

$$\tau_c = (Gbd/4\pi w_e^2) \{ 1 + 2(d/w_e) \}$$

where  $G$  is the shear modulus,  $d$  is the period length of the Peierls potential and  $w_e$  is the equilibrium separation between partials. It can be seen that the critical stress varies roughly as the inverse of the square of dissociation distance. Recalling the observed decrease in dissociation distance upon the addition of Ni to Fe-40Al, this model is qualitatively consistent with the increase in the nearly plateau-like yield strength and the observed line orientation of the dislocations. The predicted increase in critical stress is however significantly less than that observed, particularly between Fe-40Al and 50Fe-10Ni-40Al compositions. The Takeuchi model is based on continuum elasticity, and does not include possible changes to the superpartial core structures. Thus, it would appear that the observed strengthening deserves further investigation from an atomistic approach. Nonetheless, the relatively simple model of Takeuchi [25] also appears to have some merit in explaining several aspects of the strengthening effect of Ni.

The solid solution softening effect observed from Ni-40Al to 10Fe-50Ni-40Al appears to be associated with the replacement of Ni antisite defects with Fe antisite defects on the Al sublattice in this composition range. Pike [6] has argued that considering the Goldschmidt radii of 1.24, 1.27, and 1.43 for Ni, Fe, and Al respectively, then replacing Fe atoms with Ni atoms actually reduces the size misfit on the Al sublattice. That this regime is controlled by more classical solute (or anti-site defect) interactions is indeed consistent with the dominance of edge dislocations in the Ni-rich compositions. This argument is also supported by a significant increase in lattice parameter with Fe additions. There are however several aspects of this behavior which remain difficult to reconcile on the basis of this explanation. First, the softening effect and observed lattice parameter increase is more dramatic than might be expected on the basis of the modest size misfit between Ni and Fe atomic radii. In addition, the measured yield strength for 10Fe-50Ni-40Al is slightly *lower* than that for Ni-50Al, based on previous measurements [26]. The recent work of Liu, et al [27] exploring magnetic effects on atomic

size in these compounds appears to offer important, additional insight with regards to the magnitude of the softening due to Fe additions.

Only a preliminary explanation can be offered at this time for the composition dependence of the yield strength variation with temperature. With the exception of the 30Fe-30Ni-40Al composition, the transition from  $a\langle 111 \rangle$  glide to  $a\langle 100 \rangle$  climb appears to occur at about 700K. It should be noted that we have not yet determined the yield strength and microstructure for 30Fe-30Ni-40Al at 700K, however the knee clearly lies somewhere between 600K and 775K. Thus, a similar “knee” temperature may be exhibited by all of the ternary compositions, in spite of the radically different CRSS values for  $a\langle 111 \rangle$  activity at lower temperatures.

These results indicate that the most significant improvement in strength at intermediate temperatures is offered by Ni antisite defects in the Ni-rich, binary compounds. The “knee” is delayed to significantly higher temperatures in comparison to all of the ternary compositions, which accounts for the higher strength in this regime. In Ni-44Al, it has been conclusively shown that the “knee” is associated with a decomposition of  $a\langle 111 \rangle$  dislocations into  $a\langle 110 \rangle$  and  $a\langle 001 \rangle$  dislocations [20], and that  $a\langle 110 \rangle$  dislocation glide processes dominate above the “knee” [28]. In contrast, in the ternary compounds,  $a\langle 100 \rangle$  climb appears to dominate, even at temperatures just above the “knee”. In this respect, the mechanism of the slip transition in the ternary compounds appears to be similar to that observed in binary, stoichiometric Ni-50Al. The position of the “knee” and the deformation processes at higher temperature should be strongly affected by vacancy content and mobility, since both  $a\langle 100 \rangle$  climb and  $a\langle 110 \rangle$  “glide” are diffusion mediated processes (the latter involving coordinated climb/glide of coupled  $a\langle 100 \rangle$  dislocations, as described in detail elsewhere [28-30]). The importance of vacancy kinetics is emphasized by considering the recent work of Collins and co-workers [31]. Using the perturbed-angular correlation of gamma rays (PAC) method, they have determined that vacancies become mobile in Ni-50Al at about 623°C, which corresponds very well with its “knee” temperature. This favorable correlation suggests that a complete understanding of the effect of composition (including ternary additions) on the slip transition, the “knee” temperature and the deformation processes at higher temperature will require additional understanding of vacancy content and kinetics in these alloys.

## 5. CONCLUSIONS

The variation of yield strength with composition for  $\langle 100 \rangle$  oriented, B2 single crystals in the  $\text{Fe}_x\text{-Ni}_{60-x}\text{-Al}_{40}$  pseudobinary system has been correlated with dislocation behavior at both room temperature and elevated temperatures. At room temperature, the yield strength varies in a complex way with composition, indicating complicated solute effects on the CRSS for  $a\langle 111 \rangle$  dislocations, which dominate the deformation microstructures for all compositions at lower temperature. Dramatic solute strengthening is observed for Fe-rich compositions which can not be rationalized on the basis of classical solute strengthening theory alone. Additional factors may include an increase in the elastic modulus and the APB energy in this composition range. The possible relationship between the CRSS for  $a\langle 111 \rangle$  superdislocation motion and APB energy has been discussed in the context of a model by Takeuchi [25] for uncorrelated kink-pair formation on the  $a/2\langle 111 \rangle$  superpartials. This explanation is also consistent with the predominance of the screw line direction for compositions up to 30Fe-30Ni-40Al, which exhibits the highest yield strength of all the ternary compositions. The solute softening observed between 60Ni-40Al and 10Fe-50Ni-40Al is explained based on the preference of Fe to occupy the Al sublattice, thereby reducing the size misfit on this sublattice. Consistent with this view, the  $a\langle 111 \rangle$  dislocations tend to align preferentially along edge line directions in this composition range, indicating that the elastic interactions with substitutional solutes or anti-site defects are important. However, the magnitude of the softening is difficult to justify based on the small difference in atomic radius between Ni and Fe. At elevated temperatures, all compositions exhibit a “knee” in the yield strength, above which rapid softening occurs. For all the ternary compositions studied to date, this softening is related to a transition to  $a\langle 100 \rangle$  climb activity above the “knee”.

## ACKNOWLEDGEMENTS

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