Yielding Behavior of a γ''-Precipitation Strengthened Co Ni Cr Nb Fe Alloy

M. C. CHATURVEDI AND D. W. CHUNG

The yielding behavior of a 35 Co, 35 Ni, 15 Cr, 12 Nb, 3 Fe alloy, precipitation strengthened by an ordered bct γ'' phase has been studied. It was observed that during the early stages of aging at 973 K γ'' precipitation particles are sheared by the glide dislocations which occur in pairs. During this stage both order as well as coherency strain hardening mechanisms seem to be operative. Upon continued aging, when γ'' phase starts to transform to a stable ordered orthorhombic β phase, this alloy does not lose strength but its ductility is reduced. The strengthening due to β phase is attributed to the dislocation bypassing mechanism.

THE strength of many nickel base alloys is attributed to the formation of γ'' phase during the aging heat treatment.[1-8] It has been shown, conclusively, that the γ'' phase is based upon Ni_3Nb and has a DO_{22} type ordered bct structure with a c/a ratio of about 2.04. This phase precipitates coherently on {100} planes of the fcc matrix and has a disc shape with the C-axis perpendicular to the discs. Upon continued aging metastable γ'' phase transforms to a stable orthorhombic β-Ni_3Nb phase which results in a decrease in strength. Since γ'' phase is similar to γ' phase *i.e.*, it has an ordered structure and precipitates coherently, the strengthening mechanisms could also be similar to those observed in γ' precipitation hardened alloys *i.e.*, order strengthening and/or coherency strain strengthening. In Inconel 718, precipitation hardened by γ'', Oblak *et al*[5] have found coherency strengthening to be the principal mechanism. However, they also concluded that the ordered structure of the γ'' phase is important as deformation occurs by pairs of a /2 {110} dislocations. In a previous study on Co Ni Cr alloys containing Nb and Fe the main precipitating phase was found to be γ'' with similar precipitating characteristics as those observed in Inconel 718.[8]

It was also observed that, unlike Inconel 718, the hardness of 35 Co, 35 Ni, 15 Cr, 12 Nb, 3 Fe (wt pct) alloy remained unchanged although the metastable γ'' phase had completely transformed to the stable orthorhombic β phase. In the present study the influence of microstructures on the precipitate: dislocation interaction and its relation to the yield strength are discussed to obtain an understanding of the strengthening mechanisms operating in this alloy.

EXPERIMENTAL TECHNIQUES

650 gm melts of the alloy were prepared in an induction melting furnace in an argon atmosphere using 99.99 pct pure alloying elements except Nb which was 99.95 pct pure. The melts were cast into 20 mm square ingots and homogenized at 1423 K for 3 days. The ingots were then cold rolled to 1.0 mm thick strips with frequent intermediate anneals. These strips were used to produce $1.0 \times 5 \times 20$ mm gage length flat tensile specimens. The tensile specimens were given a final solution treatment at 1423 K, quenched in ice water and aged at 923 K to various lengths of time. All the heat treatments were carried out in argon-filled vycor capsules and were terminated by quenching in ice water. The structure of aged as well as aged and deformed specimens was examined by thinfilm electron microscopy. Thin films were prepared from 125 μm thick strips using an electrolytic jet polishing technique with 5 pct perchloric acid − 95 pct methanol bath at 223 to 233 K. The precipitate size was measured from areas in the {100} matrix orientation only. The volume fraction of precipitate was determined by electrolytic extraction in 1 pct citric acid − 1 pct ammonium sulphate solution at 1.5 V.[9] Since the solution-treated samples also contained some undissolved particles, the volume fraction of precipitate was taken to be the difference between the amount of particles extracted from the aged and the amount from solution treated specimens.

RESULTS

It was reported earlier[8] that in this alloy during the early stages of aging at 973 K, the main precipitating phase was observed to be ordered bct γ'' phase. This phase precipitates coherently on {100} planes of the fcc matrix and has a disc shape with the C-axis being perpendicular to the discs. The tetragonal distortion due to coherency strains was 0.027 and its volume fraction after 17 min of aging was found to be 11.9 pct. The variation in the values of volume fraction in specimens aged for 50 min, 1.5, 3 and 15 h was observed to be between 11.9 to 12.5 pct. Therefore, it is assumed that most of the precipitation is complete after about 17 min of aging and average volume fraction is 12.0 pct. The particle size was observed to follow the Liftshitz-Wagner theory of diffusion controlled growth. During very early stages of aging it was not possible to measure the particle size accurately. Therefore, values were estimated by extrapolating the log-log plot of particle diameter vs time. After 17 h of aging γ'' precipitate starts to transform into a stable, ordered orthorhombic

M. C. CHATURVEDI is Professor, Metallurgical Science Laboratory, Department of Mechanical Engineering, University of Manitoba, Winnipeg, Canada, R3T 2N2. (On sabbatical leave with Aluminum Company of Canada, Research Centre, Kingstron, Ontario until June 30, 1981). D. W. CHUNG was formerly Research Associate in the Metallurgical Science Laboratory, Department of Mechanical Engineering, University of Manitoba, Winnipeg, Manitoba, Canada R3T 2N2.

Manuscript submitted February 22, 1980.

ISSN 0360-2133/81/0112-0077$00.75/0

β phase and in a specimen aged for the 1150 h only β phase was observed.

Tensile Test Results

The room temperature mechanical properties of aged specimens were determined by tensile tests using an Instron testing machine at an initial strain rate of 4.16 $\times 10^{-4}$ S^{-1}. The variation in 0.2 pct yield strength (YS), ultimate tensile strength (UTS) and total elongation to fracture, with aging time at 973 K is shown in Fig. 1. During the early stages of aging the variations in strength and ductility follow the normal behavior, *i.e.*, as aging time is increased the strength increases and ductility decreases. The peak strength is achieved after about 2 h of aging. Upon continued aging up to 1150 h both YS and UTS remain almost he same as those observed at the peak. However, a decrease in ductility from 18 to 7 pct is observed. This behavior is different from other γ'' strengthened alloys where the appearance of β phase results in a significant reduction in strength.[2-4,6,10] The variation in yield strength with the radius of γ'' discs is shown in Fig. 2. It observed that initially the yield strength increases linearly but after about 2 h it is almost independent of the γ'' particle size.

Deformation Structures

In order to study the dislocation interaction with γ'' precipitates, thin foils of the aged specimens were examined after 2 to 4 pct tensile deformation at room temperature. Figure 3 shows the structure of a specimen aged for 17 min and deformed 4 pct. The radius of γ'' discs in the specimen is estimated to be about 33Å and dislocations are seen to occur mostly in pairs as was observed in Inconel 718.[9] Another example of a similar dislocation structure is shown in Fig. 4 which is the structure of a specimen aged for 50 min at 973 K and deformed 3.5 pct. The radius of γ'' at this stage is estimated to be 48Å and almost all dislocations are seen to occur in pairs. Although γ'' precipitate particles are not visible in the micrographs, their presence is suggested by the mottled nature of the structure and the presence of γ'' superlattice spots in the electron diffraction pattern. A steep rise in the yield strength of the specimens also confirms the presence of γ'' precipitate particles. The occurence of paired dislocations suggests that the ordered γ'' precipitates are being sheared by the glide dislocations as observed in γ' precipitation hardened alloys.[11,12]

Upon further aging, it was unfortunaely not possible to resolve glide dislocations due to coherency strain fields around γ'' particles. However, sheared particles could be observed by dark field techniques. An example is shown in Figs. 5(*a*) and (*b*) which are the bright and dark field micrographs, respectively, of a specimen aged for 120 h and deformed 3 pct. At this stage of aging the γ'' particle radius is about 250Å and a large numer of particles seems to have been sheared along the broad slip bands (Fig. 5(*b*)). In this specimen a significant amount of β phase was also present which appears not to have sheared when the specimens were deformed by a small amount. However, when this specimen was

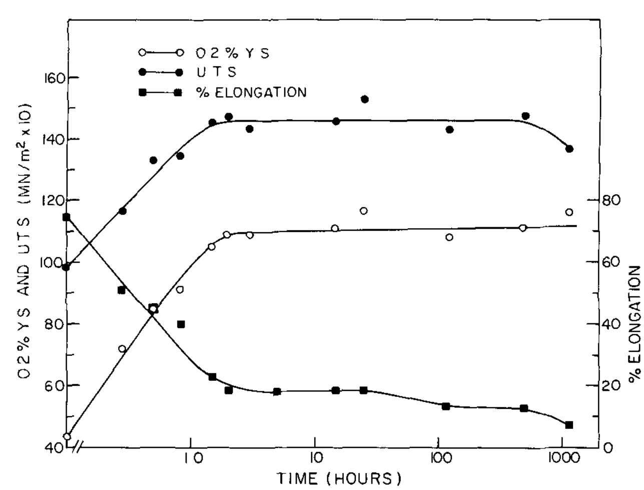


Fig. 1—Effect of aging at 973 K on the tensile properties.

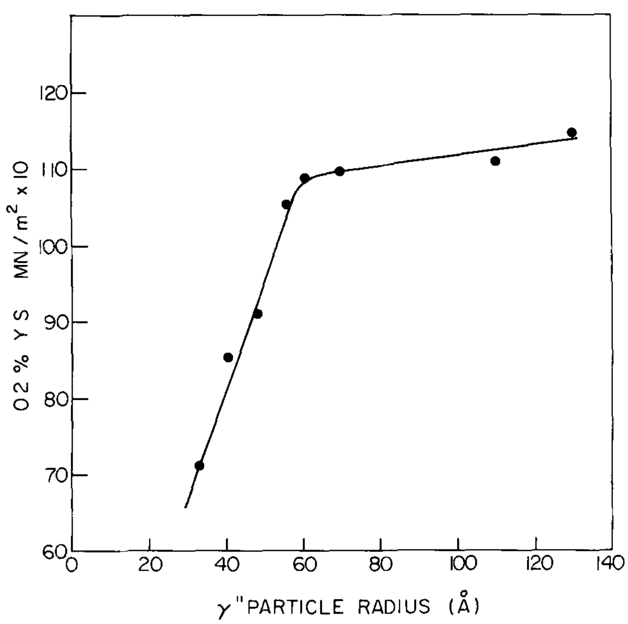


Fig. 2—Effect of γ'' particle size on 0.2 pct offset yield strength.

Fig. 3—Structure of a specimen aged for 17 min and 4 pct deformed.

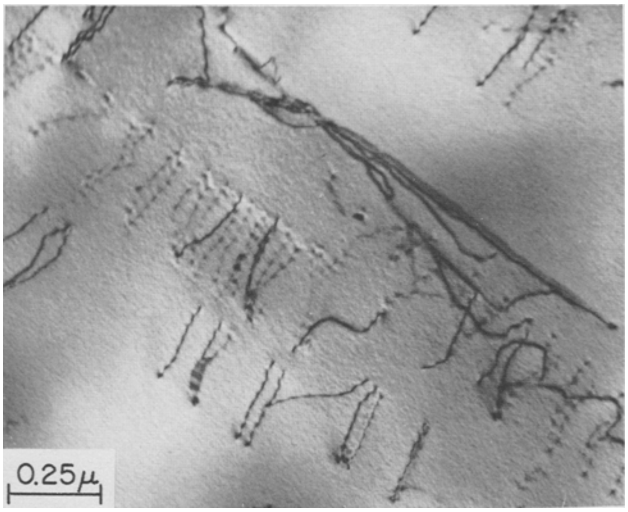
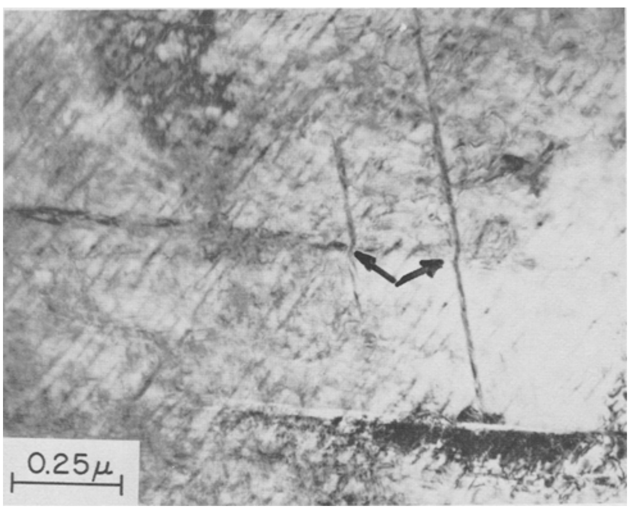
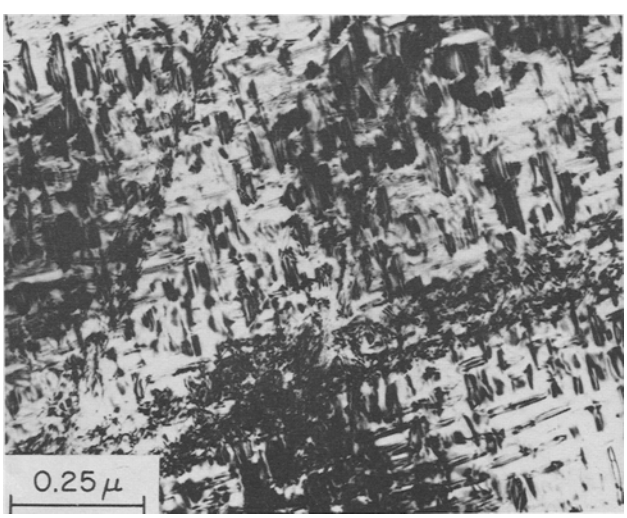


Fig.4—Structure of a specimen aged for 50 min and 4 pct deformed.



(a)



(b)

Fig. 5—(a) Bright field structure of a specimen aged for 120 h and deformed 4 pct. (b) Dark field structure of Fig. (a) taken with a γ'' precipitate reflection.

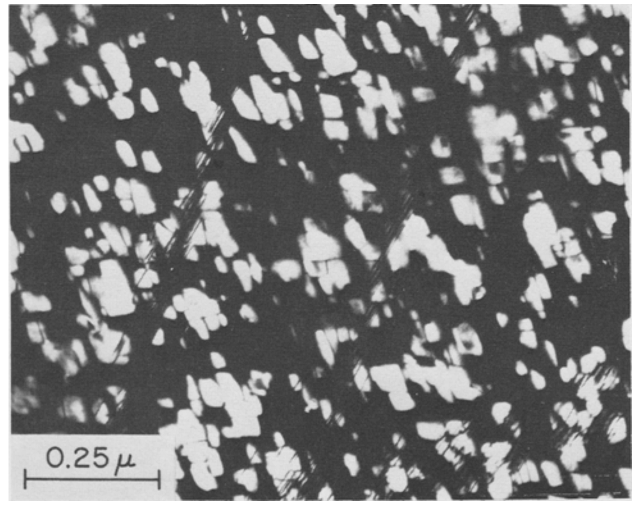


Fig. 6—Structure of a specimen aged for 120 h and deformed about 8 pct by rolling.

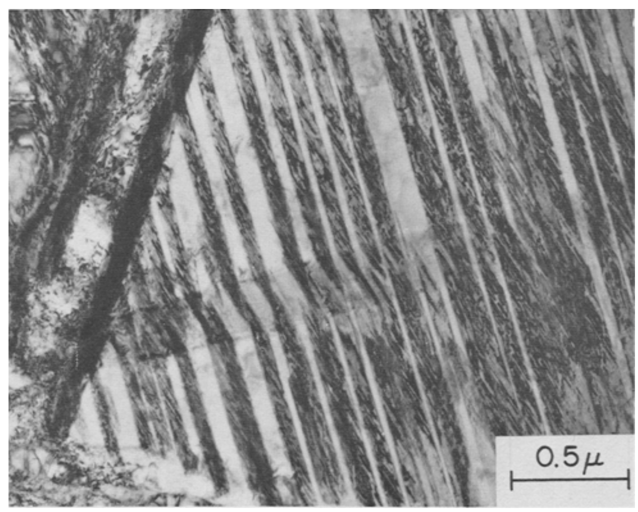


Fig. 7—Structure of a specimen aged for 1150 h and deformed 10 pct by rolling.

deformed 8 pct by rolling occasionally a few sheared laths of β phase were observed as shown in Fig. 6. Occasionally β phase was also sheared by deformation twins as shown in Fig. 7 which is the structure of a specimen aged for 1150 h and deformed 10 pct by rolling.

None of the samples examined, from very early stages to 1150 h of aging, showed dislocation loops around γ'' particles which have been observed around large γ' particles in other alloys, although it is possible that the presence of high coherency strains around γ'' particles obscured the presence of such loops.

DISCUSSION

The above results show that during the early stages of aging γ'' is the main precipitating phase. The presence of γ'' increases the yield strength of the alloy by 150 pct over the solution treated condition and maximum strength is obtained in relatively short aging times. During this stage of precipitation, γ'' particles are sheared by glide dislocations (Figs. 3 and 4) and they move in pairs due to the ordered structure of the precipitates. In underaged γ' precipitation hardened alloys where the ordered γ' phase also precipitates coherently, a similar precipitate dislocation interaction has been observed. A great deal of controversy exists regarding the relative contribution made by coherency strain and order strengthening mechanisms to the strength of γ' strengthened alloy. However, recently it has been recognized that if a significant mismatch exists between γ'' andγ' phases both the mechanisms should play a role in determining the overall strength of the alloy,[11,12] and it has been found that in Nimonic 80 where γ/γ' mismatch is 0.35, both the mechanism contribute almost equally.[12] The strengthening mechanisms in the underaged γ'' precipitation hardened alloys should be similar to those observed in γ' precipitation hardened alloys because γ'' is not only an ordered phase which precipitates coherently, but is also sheared by a pairwise motion of glide dislocations. However, in Inconel 718, Oblak, *et al*[5] found that the strengthening was mainly due to the coherency strain and the significance of order in γ'' particles was to ensure a pairwise motion of dislocation. This may well be because of a significantly larger value of misfit in γ/γ'' alloys, 0.0286, as compared to γ/γ' alloys where it is generally between 0.0035 to 0.01. To determine if in the present γ/γ'' alloy strengthening was also mainly due to coherency strains only the theoretical values of increased in CRSS due to the precipitation of γ'' were calculated by the same method used for Inconel 718.[5]

The increment in CRSS due to coherency strains when γ'' particles are sheared by an edge dislocation, $\Delta\tau_{coh}$ is given by:[13]

$$\Delta\tau_{coh} = 1.7\,\mu\,|\epsilon|^{3/2}\left[\frac{h^2 f_v(1-\beta)}{2\mathbf{b}R}\right]^{1/2} \qquad [1]$$

and due to order strengthening, $\Delta\tau_{ord}$, when shearing of γ'' takes place by a pairwise motion of dislocations is given by:[5]

$$\Delta\tau_{ord} = \left[\frac{\gamma APB}{2\mathbf{b}}\right]\left[\left(\frac{4\gamma APB\,fv}{\Pi T}\left[\frac{\sqrt{6}Rh}{3}\right]^{1/2}\right)^{1/2} - \beta f v\right] \qquad [2]$$

where μ is the shear modulus, ϵ is the tetragonal misfit, fv is the volume fraction, R is the radius and h is the half thickness of γ'' precipitates, **b** is the Burgers vector of the dislocation, γAPB is the antiphase boundary energy of γ'' and T is the line tension of the dislocation. The value of constant β depends upon the distribution of γ'' and is equal to 1/3 when they are present along all the three invariants as observed in the present investigation. By assuming the values of γAPB and μ to be similar to those observed in Inconel 718, the theoretical values of $\Delta\tau_{coh}$ and $\Delta\tau_{ord}$ were calculated for various aging times up to the peak strength. The actual con-

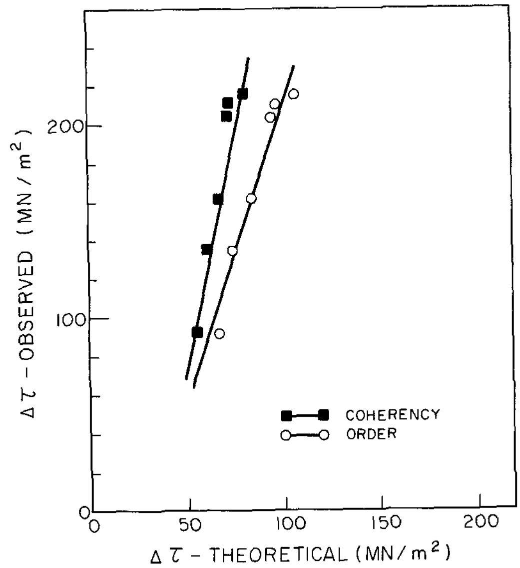


Fig. 8—Plots of observed *vs* theoretical values of $\Delta\tau$ during early stages of aging.

tribution of precipitation strengthening to the yield stress was determined by $\sigma_y - \sigma_o$, where σ_y and σ_o are the 0.2 pct offset yield stress of the aged and solution treated specimens, respectively. The increase in CRSS was then calculated by dividing $(\sigma_y - \sigma_o)$ by 3.1, the Taylor factor for fcc materials. The observed values of $\Delta\tau$ are plotted against the theoretical values of $\Delta\tau$ by both coherency and order strengthening, as shown in Fig. 8. It is seen that each of the two mechanisms underestimate the increase in CRSS by about half of the observed values. Although it is not expected to obtain an exact correspondence between the theoretical and the observed values, it can be concluded that in this alloy, up to the peak level, both coherency as well as order strengthening due to γ'' are responsible for the observed increase in strength.

Figure 1 also shows that upon continued aging up to 1150 h the strength of this alloy remains almost constant despite the transformation of γ'' phase to a stable orthorhombic β phase. This transformation begins after 15 to 25 h of aging and only β phase is present in a specimen aged for 1150 h. This would suggest that any softening due to the growth as well as transformation of γ'' phase is compensated by the strenthening due to the formation of a β phase whose interparticle spacing continues to decrease and volume fraction continues to increase as increasing amounts of γ'' phase are transformed. At small strains, in the presence of γ'', it was not possible to examine individual dislocations due to the presence of large coherency strains, however, β particles were not observed to be sheared by deformation. When only β phase was present, a small amount of deformation only produced dislocation pile ups at the precipitate matrix interphase. Therefore, strengthening due to β phase can be attributed to the dislocation bypassing mechanism. This is consistent with the large size of β phase particles. Occasional deformation of β phase observed at large strain (Figs. 6 and 7) is due to the accumulation of stress due to pile up of dislocation at the matrix precipitate interface.

CONCLUSIONS

1) During the early stages of aging the strength of the alloy is attributed to the precipitation of an ordered bct γ'' phase which forms coherently on $\{100\}$ matrix planes.

2) Upon deformation during this stage, yielding occurs by the shearing of γ'' particles by glide dislocations which move in pairs. The strengthening during the particle shearing stage seems to be due to both order and coherency strain hardening mechanisms.

3) Upon continued aging, γ'' transforms to a stable orthorhombic β phase. Overaging of γ'' phase does not result in a reduction in strength as has been observed in other γ'' precipitation strengthened alloys. It is suggested that in this alloy β phase is also a very effective strengthening phase and compensates any loss in strength due to the overaging of γ'' phase.

4) Strengthening by the β phase particles is attributed to the dislocation bypassing mechanism.

ACKNOWLEDGMENTS

The authors would like to thank Dr. J. R. Cahoon of the Department of Mechanical Engineering, University of Manitoba, for many useful discussions and Sherrit-Gordon Mines, Fort Saskatchewan, Alberta, Canada, for supplying the alloying metals. Financial support from the National Science and Engineering Research Council, Ottawa, Canada, is also greatefully acknowledged.

REFERENCES

1. P. S. Kotval: *Trans. TMS-AIME*, 1968, vol. 242, p. 1764.
2. I. Kirman: *J. Iron Steel Inst.*, 1969, vol. 207, p. 1612.
3. D. F. Paulonis, J. M. Oblak, and D. A. Duvall: *Trans. ASM* 1969, vol. 62, p. 611.
4. I. Kirman and D. H. Warrington: *Met. Trans.*, 1970, vol. 1, p. 2667.
5. J. M. Oblak, D. F. Paulonis, and D. S. Duvall: *Met. Trans.*, 1974, vol. 5, p. 143.
6. V. Ramaswamy, P. R. Swann, and D. R. F. West: *J. Less-Common. Met*, 1971, vol. 27, p. 17.
7. R. Cozar and A. Pineau: *Met. Trans.*, 1973, vol. 4, p. 47.
8. M. C. Chaturvedi and D. W. Chung: *Met. Trans. A.*, 1979, vol. 10A, p. 1579.
9. O. H. Kreige and J. M. Baris: *Trans. ASM*, 1969, vol. 62, p. 195.
10. M. Raghavan: *Met. Trans. A.*, 1977, vol. 8A, p. 1071
11. E. J. Lee and A. J. Ardell: *Proc. 5th Int. Conf. on Strength of Metals and Alloys*, vol. 1, p. 633, Aachen, 1969.
12. A. Melander and P. A. Persson: *Met. Sci.*, 1978, vol. 12, p. 391.
13. J. M. Oblak, D. S. Duvall, and D. F. Paulonis: *Mater. Sci. Eng.*, 1974, vol. 13, p. 51