# GEORGIA INSTITUTE OF TECHNOLOGY
### OFFICE OF RESEARCH ADMINISTRATION

## RESEARCH PROJECT INITIATION

Date:  May 23, 1973

Project Title:  A Study of the Structure and Mechanical Properties of Ordered Alloys

Project No:  E-19-617

Principal Investigator  Dr. E. A. Starke and Dr. B. G. LeFevre

Sponsor:  AEC - Oak Ridge Operations

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RA-3 (6-71)

# GEORGIA INSTITUTE OF TECHNOLOGY
## OFFICE OF RESEARCH ADMINISTRATION

## RESEARCH PROJECT TERMINATION

Date: **May 13, 1974**

Project Title **A Study of the Structure & Mechanical Properties of Ordered Alloys**

Project No: **E-19-617**

Principal Investigator: **Dr. E. A. Starke & Dr. B. G. LeFevre**

Sponsor: **AEC – Oak Ridge Operations**

Effective Termination Date: **3-31-74 (Contract Expiration)**

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RA-4 (5/70)

# Structure and mechanical properties of stress-ordered Ni$_4$Mo

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## Summary

The ordering of Ni$_4$Mo by isothermal annealing with and without an applied stress at 700°C has been studied. The microstructural features developed during ordering have been correlated with the mechanical properties. Ordering under an applied stress affects both the kinetics and the number of c-axes variants which exist within any one grain. The number of variants has an influence on the mechanical behavior.

## 1. INTRODUCTION

Ni$_4$Mo exhibits a face-centered cubic disordered structure ($\alpha$ phase) above the order—disorder transformation temperature, 868°C[1], and forms a body-centered tetragonal ordered structure ($\beta$ phase) below this temperature. The ordering reaction is accompanied by a 1.19% contraction in the c-axis of the b.c.t. structure[2] and a rotation of arctan (1/3) degree with respect to the f.c.c. structure. The c-axis of the superlattice is always parallel to one of the three original axes of the f.c.c. structure, and for each c-axis there are two different rotational angles possible. Therefore, six different orientations or domains, relative to the disordered lattice, may occur. For a normal ordering condition, all six are equally distributed in the crystal except in the neighborhood of a free surface[3].

Three kinds of domain boundaries may form during ordering. The first is formed by impingement of two domains with the same orientation (parallel c-axes, and same sign rotation angle); the second is formed by two domains with different rotation angles but with parallel c-axes; and the last one is formed by two domains with orthogonal c-axes. These domain boundaries are called anti-phase-domain boundaries (APDB), antiparallel-twin boundaries (APTB), and perpendicular-twin boundaries (PTB), respectively. In general, the APDB has the lowest energy of the three since no appreciable distortion occurs except when a Mo—Mo relationship exists across the boundary. The APTB usually contains small strains due to the irregular distribution of Mo atoms in the boundary. The PTB has the highest energy of all three since the orthogonality of the c-axes of the domains creating the boundaries causes large distortions in the boundaries[3].

X-Ray line broadening analysis has shown that the microstrains which develop during isothermal ordering at 700°C and 650°C are proportional to the tetragonality of the b.c.t. superlattice structure and also dependent on the degree of order and domain size[4]. The microstrains are primarily a result of the strain at the PTB produced by the lattice misfit during ordering. Generation of dislocations at the PTB may be possible during the late stages of ordering[3]. This has been verified indirectly by the internal friction studies of Ling, Collado and Starke[5].

Both APDB's and PTB's affect the work hardening coefficient[6], but the presence of PTB's has a more significant influence on the

yield strength[7] since microstrains are present at the coherent PTB's. A quantitative analysis of the effects on mechanical properties by these two kinds of boundaries is possible if the fractions of either one can be controlled. For instance, if all c-axes are parallel there will be no PTB. Hirabayashi[8] investigated the influence of a small stress applied during the ordering anneal on the dimensional change accompanying ordering of CuAu and found that a change in distribution of domain orientation had been effected by the stress. He also verified that an applied compressive stress will favor the development of that domain orientation which most effectively contracts the crystal along the compressive axis. Arunachalan and Cahn[9] ordered CuAu single crystals under a compressive stress and were able to orient approximately 90% of the c-axes in the direction of the compressive stress. The 1.19% contraction that occurs along the c-axis during ordering of $Ni_4Mo$ suggests that this alloy might be amenable to stress ordering.

The present study was undertaken to quantitatively characterize the microstructural features, *i.e.* degree of order, domain size and type, produced by ordering $Ni_4Mo$ with and without an applied stress and to correlate these features with the mechanical behavior.

## 2. EXPERIMENTAL METHODS

$Ni_4Mo$ alloys were prepared by arc melting high purity nickel and molybdenum on a water-cooled copper hearth under an argon atmosphere. Each alloy was melted eight times to insure adequate mixing of the nickel and molybdenum. The ingots were homogenized in an evacuated quartz tube at $1000°C$ for one week, quenched in iced water, swaged to a diameter of 0.2 centimeter and cut to lengths of 8 centimeters. They were then heat treated in an evacuated quartz tube at $1000°C$ for 24 hours to remove the stresses caused by the mechanical deformation, quenched in iced water and chemically polished. Chemical polishing was carried out at $90°C$ in a solution of 225 ml nitric acid, 150 ml sulfuric acid, 100 ml distilled water and 3 grams of NaCl.

Two specimens, one for stress-free ordering and another for stress ordering, were inserted in quartz tubes and heat treated simultaneously in the center of the aging furnace for each

ordering condition. Each stress ordered specimen was compressed by a stainless steel rod with a free weight of about 11 kilograms, so that the compressive force would be constant during heat treatment. In order to prevent diffusion between the contacts of the stainless steel (the rod and the base) and the stress ordered specimen, two extra pieces of $Ni_4Mo$ alloy were placed at both ends of the specimen within the quartz tube. The specimen under stress was placed as close as possible to the stress-free specimen so that the heat-treating conditions, such as time, temperature and environment, would be the same for both during ordering. The specimens were ordered for various times by isothermal aging at $700°C$. The temperature and times were chosen from previous experience with $Ni_4Mo$ polycrystalline[10] and single crystal samples[3]. All heat treatments were conducted under vacuum ($10^{-5}$ Torr) using the vacuum chamber of an MRC model EVD-97 unit.

The degree of order after various heat treatments was determined from Debye—Scherrer patterns by measuring the integrated intensity with a microphotometer and applying the method of LeFevre and Starke[11]. The order parameter was measured on each tensile sample prior to testing. The average domain size (which includes all three types of domains) was also determined by X-ray diffraction using Scherrer's equation. The $(110)_T^*$ superlattice peak was used for these calculations and was corrected for strain and instrumental broadening by comparison with the nearby $(211)_T$ fundamental peak.

The tensile tests were conducted on an Instron testing machine using a 2.54 cm extensometer and a strain rate of 0.05 per minute. All specimens were pulled to fracture. Microhardness measurements were made on undeformed samples with a Knoop diamond indenter and a 500 gram load. Samples for electron microscopy studies were cut from the tensile samples prior to testing and electropolished using standard techniques[12]. The thin foils were examined in transmission at 100 kV. Two samples, stress and stress-free ordered after 3000 minutes, were mechanically polished and then chemically or electrically polished. The polarized light photomicro-

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* Subscript T refers to the tetragonal indices; cubic indices are undesignated.

graphs of those two samples were taken with a Zeiss microscope.

## 3. RESULTS

The structure and mechanical properties of $Ni_4Mo$ were studied after isothermal ordering, with and without an applied stress at 700°C. The degree of long-range order, average domain size and microstructure were correlated with the tensile and hardness measurements which provided an indication of the strength and work hardening of the alloy at various stages of ordering.

The average domain sizes ($D$) of two sets of samples, stress-free and stress ordered, after various ordering times ($t$) are shown in Fig. 1 by the characteristic linear relation of the ln $D$ versus ln $t$ plot with a slope of $1/(2.8)$. The domain size of the stress-ordered sample is larger than that of the stress-free ordered sample after the same period of ordering time. The values of the domain size, long-range order parameter ($S$), hardness, yield stress and work-hardening coefficient, for the stress and stress-free ordered samples after various ordering times, are given in Table 1. The work-hardening coefficients were measured at 2% strain from the stress—strain curve. All parameters increase faster with aging time for the stress-ordered samples than for the stress-free-ordered samples. However, the stress-free-ordered samples have higher maximum values of yield stress and work hardening coefficient, and "over-aging" is more pronounced for the times used in this study.

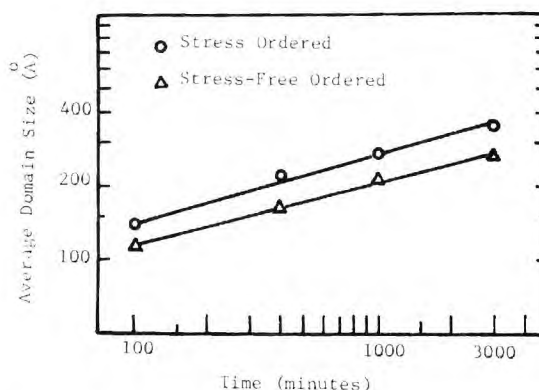The normals to all the foils examined in the electron microscope were approximately par-



Fig. 1. Variation of the average domain size of $Ni_4Mo$ with aging time at 700°C.

allel to the stress axis of the stress-ordered samples. All types of domain boundaries were found in both stress-ordered and stress-free-ordered samples. However, perpendicular twins were much more frequently observed in stress-free-ordered samples than in stress-ordered samples. The following micrographs are designed to explain the different domain structures present in the late ordering stage in $Ni_4Mo$. The identification of the boundaries is based on the argument of previous workers[13,14].

Because of the contraction and expansion along the axes, the possible coherent planes for anti-parallel twins are (100), (010), (110) and ($1\bar{1}0$) since the d spacing remains the same on both sides of the twin boundary. Similarly, the preferred planes for perpendicular twin boundaries are (101) and ($10\bar{1}$)[13]. In the early stage of ordering, when $S$ is small, and there has been little or no change in lattice constants, APT and PT boundaries are not necessarily on preferred planes and may

TABLE 1

| Aging time (minutes) | LRO parameter | | Domain size (Å) | | Hardness (Knoop) | | Yield stress (kg/cm$^2$) | | Work hardening coefficient $\times 10^3$ (kg/cm$^2$/% strain) | |
|---|---|---|---|---|---|---|---|---|---|---|
| | SFO* | SO** | SFO | SO | SFO | SO | SFO | SO | SFO | SO |
| 0 | 0 | 0 | — | — | 228 | 228 | 3500 | 3500 | 1.10 | 1.10 |
| 100 | 0.75 | 0.82 | 114 | 140 | 394 | 422 | 4680 | 5160 | 1.29 | 1.42 |
| 400 | 0.95 | 1.00 | 161 | 221 | 455 | 442 | 6500 | 6350 | 1.64 | 1.60 |
| 1000 | 1.00 | 1.00 | 215 | 270 | 486 | 443 | 7500 | 6320 | 1.89 | 1.62 |
| 2000 | — | — | — | — | 475 | 436 | 7050 | 6330 | — | — |
| 3000 | 1.00 | 1.00 | 266 | 355 | 455 | 438 | 6900 | 6200 | 1.61 | 1.49 |

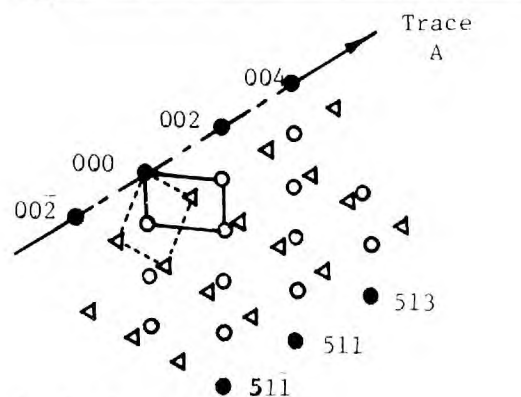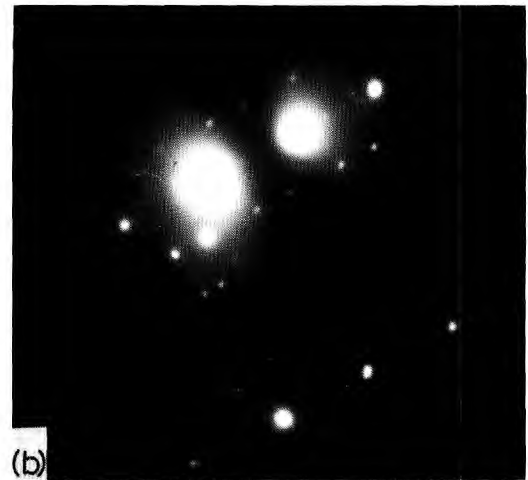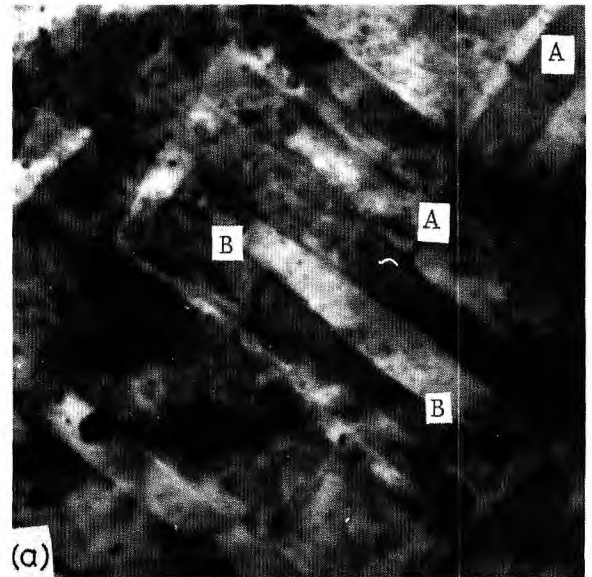\* SFO = Stress-free ordered
\*\* SO  = Stress ordered.

Fig. 2. Dark field image with a superlattice reflection showing APT's and APB's in $Ni_4Mo$ after 400 minutes stress-free ordering at 700°C (50,000X).

appear curved. As the LRO increases the lattice misfit across twin boundaries increases and PT boundaries become more planar as the lower lattice misfit planes become preferred as boundaries. Anti-parallel twin boundaries, which have lower energy than PT boundaries, may remain curved or may become planar depending on the domain's configuration and distribution. The curved APT boundaries can be seen in Fig. 2. Planar APT boundaries are observed in Fig. 3(a) which is a micrograph of a sample stress-ordered for 1000 minutes at 700°C. The diffraction pattern from the area shown in Fig. 3(a), Fig. 3(b), shows that two variants with the same c-axes are twin related. Trace analysis suggests that trace A—A may be parallel to (110)*, (010) or (100) planes, and trace B—B may be parallel to (001), (011), (01$\bar{1}$) or (00$\bar{1}$) planes. This suggestion is based on the assumption that these planes are preferred as APT boundaries[13].

Figure 4(a) shows perpendicular twin boundaries present in the stress-free-ordered sample after 1000 minutes at 700°C. A comparison of the diffraction pattern from the area shown in Fig. 4(a), Fig. 4(b), with a reciprocal lattice model containing all variants, showed that the pattern results from two vari-
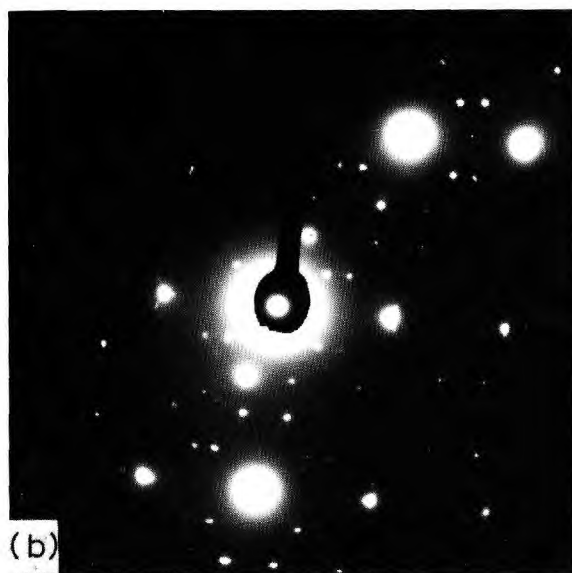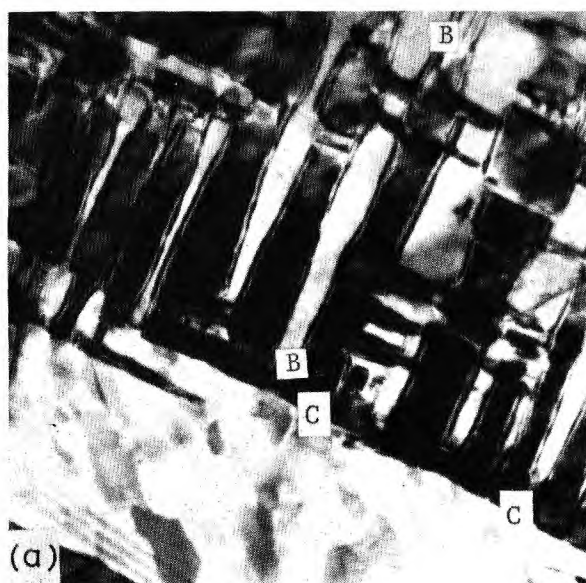
*The trace analysis is based on the cubic structure and it is not possible to distinguish between (101) and (110) planes.





Fig. 3. $Ni_4Mo$ stress ordered for 1000 minutes at 700°C.

(a) Bright field electron micrograph, [150] orientation; antiphase parallel twin structures (22,000X).

(b) Diffraction pattern from area shown in (a) (cubic indices are used).

●   Fundamental Spots
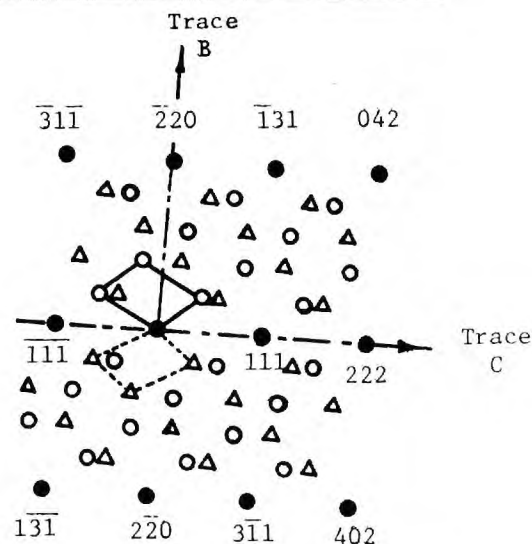○   Superlattice Spots of Variant I
△   Superlattice Spots of Variant II

Fig. 4. $Ni_4Mo$ stress-free ordered for 1000 minutes at 700°C.
(a) Bright field electron micrograph, [$\bar{1}12$] orientation; perpendicular twin structure (40,000X).
(b) Diffraction pattern from area shown in (a) (cubic indices are used).

ants of mutually perpendicular c-axes. A trace analysis suggests that the boundaries, B—B and C—C, may be parallel to {101} planes as previously suggested.

The perpendicular twin boundary may become partially incoherent after long ordering times because of the misfit strain in the perpendicular twin plane. Therefore, after the twin boundaries are eliminated by domain growth, the dislocations generated from perpendicular twin boundaries are left within the new domains and form subgrain boundaries as shown in Fig. 5. This argument is supported by the fact that these subgrain boundaries are normal to ⟨101⟩ poles, and {101} planes are the preferred planes for perpendicular twin boundaries.

The electro-polished samples revealed de-

tails in the polarized light micrographs of the domain structure while the chemically polished sample revealed the incoherent boundaries, such as grain boundaries and incoherent domain boundaries. Photomicrographs of electro-polished stress-ordered and stress-free-ordered samples are shown in Figs. 6(a) and 6(b) respectively. Since stress ordering eliminates one or two orientation variants there are fewer domain variants in Fig. 6(b) than in Fig. 6(a).

Figure 7 shows a chemically polished sample after stress ordering. It is noticed that most grains are dominated by only two domain variants. The plate-like domains are probably perpendicular twins since the perpendicular twin boundaries may become partially incoherent at the very late stage of order-

Fig. 5. Bright field electron micrograph of Ni₄Mo stress-free ordered for 1000 minutes at 700°C [011] orientation showing low angle boundaries (40,000X).
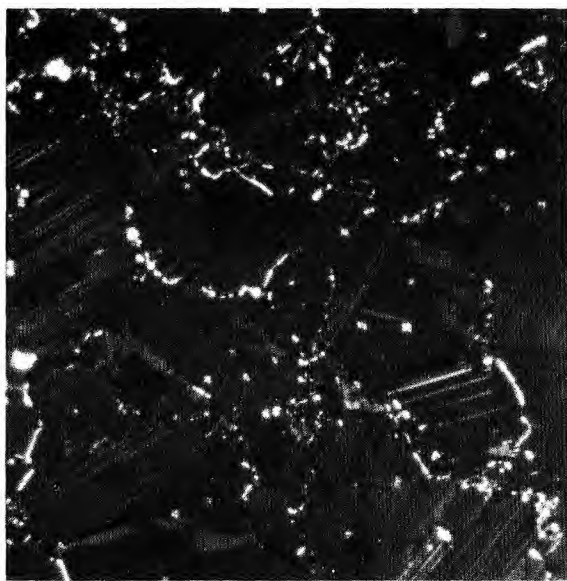


Fig. 7. Polarized light micrograph of Ni₄Mo after stress ordering 3000 minutes at 700°C. Sample was chemically polished (320X).
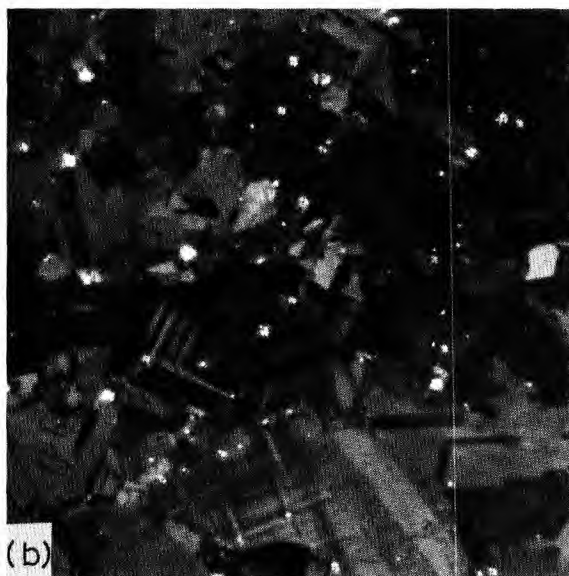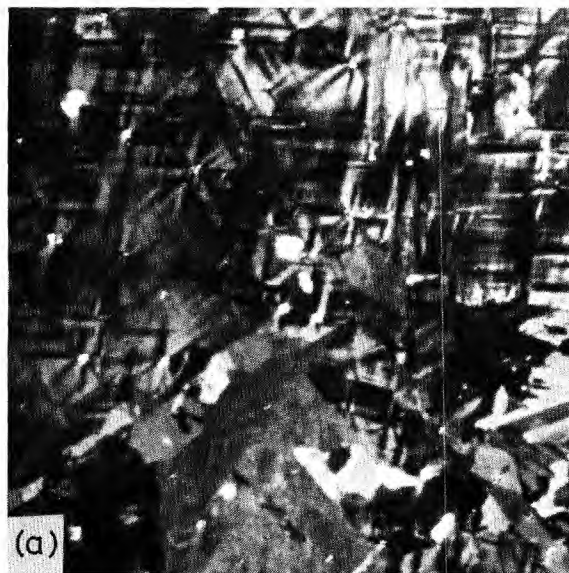


Fig. 6. The polarized light micrograph of Ni₄Mo after 3000 minutes of ordering at 700°C. Samples were electro-polished (320X).
(a) Stress-free ordered.
(b) Stress ordered.

ing as described previously. The incoherency of the domain boundaries can be revealed by chemical etching.

## 4. DISCUSSION OF RESULTS

The results of this study clearly show that ordering under an applied stress can influence the kinetics of the transformation, the microstructure and mechanical properties of Ni₄Mo. The magnitude of the influence is very orientation dependent, being paramount when the stress axis is parallel to a cube axis but is somewhat averaged in a polycrystalline

sample. Theoretically, the application of a uniaxial stress normal to a {111} oriented crystal will not alter the random morphology of the domains since the resolved stresses in the cube directions will be equal in magnitude. Pinhole X-ray patterns of samples used in this study showed them to be randomly oriented with no defined fiber texture.

Two kinds of stress-ordering effects may occur for all of the grains with favorable orientations. The first would occur if any one of the three original cube axes is close to the compression direction. In this case the c-axes of all ordered domains would be oriented in the same direction, i.e. one c-axis variant. The second would occur if one of the ⟨110⟩ poles is close to the compression direction. In this case the two closer ⟨100⟩ directions for each specific ⟨110⟩ (e.g., [100] and [010] for [110]) would be equally favored, resulting in two c-axes variants. Further orientational relationships may be obtained if the lattice twins in order to relieve ordering stresses[15,16]. Consequently, the probability of obtaining a grain containing domains with only one c-axis variant (see Fig. 6) is small, even for stress-ordered samples. In the stress-free-ordered samples, three c-axes variants normally were observed to coexist within a grain and occasionally two variants, related as perpendicular twins, were observed. Two, twin related, variants were normally observed within grains of the stress-ordered samples; however, occasionally one or three variants were observed. These features were observed by both electron and polarized light microscopy.

The kinetics of the ordering transformation was also seen to be affected by the applied stress. The applied stress tends to lower the transformation strain energy by reducing the probability of PT formation, which increases the change in free energy between the ordered and disordered state resulting in a faster ordering rate, as illustrated in Table 1. Domain growth in $Ni_4Mo$ during isothermal ordering has been described by Ling and Starke[3] by the size—time relationship $D^n - D_o{}^n = K_1 t$ where $D_o$ is the initial domain size, $D$ is the domain size at time $t$, and $n$ and $K_1$ are constants. The magnitude of $n$ has been suggested to be dependent on alloy purity, decreasing as the purity increases. A value of $n = 2.9$ was found previously for $Ni_4Mo$, and the value in the present study is very close to that, being

2.8. The average domain size for the stress-ordered samples is larger than that found for the stress-free-ordered samples for the same ordering time. This is probably due to the higher density of PT boundaries inhibiting domain boundary migration for the stress-free ordered samples.

The stress-ordered samples contained fewer PT boundaries than did the stress-free-ordered samples and, consequently, the microstrains caused by lattice misfit are lower in these samples. Therefore the yield stress and work hardening coefficients are also lower since these parameters are directly related to the microstrains[3]. The higher initial properties are simply related to the higher degree of order for the stress-ordered samples.

Ling and Starke[3] suggested that the stresses produced by the lattice misfit, in conjunction with thermal fluctuations, can generate dislocations at PT interfaces. This appears to be verified by the electron micrograph shown in Fig. 5. The extremely high yield stress of the stress-free-ordered sample after 1000 minutes of ordering can be attributed to internal strains caused by these dislocations.

## 5. CONCLUSIONS

a. Ordering under an applied stress increases the kinetics of the process owing to a reduction in the ordering stresses.

b. The stress-free-ordered samples had higher maximum values of hardness, yield strength and work-hardening rate. This is attributed to three variants existing in most grains of the stress-free-ordered samples and only two in grains of the stress-ordered samples.

c. Dislocations generated from perpendicular twin boundaries can be observed.

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L-19-617

A STUDY OF THE STRUCTURE AND MECHANICAL PROPERTIES OF ORDERED ALLOYS

Final Report

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April 1, 1969 - March 31, 1974

L-19-617

I.  Summary of Objectives and Results of Research Program.

The original objective  of the research program was· to elucidate the

various strengthening mechanisms in alloys containing both local and long

range order.  It was recognized that much of the published work relating

order and physical properties (in particular mechanical properties) was based

on incomplete or ambiguous information regarding the microstructure of order-

ed alloys.  For this reason a large portion of the work was based on a thorough

characterization of microstructural parameters of various ordering systems

subjected to thermal-mechanical treatments and the changes produced by

deformation.  Those parameters considered most important were: (i) local

order in the matrix (ii) degree of long-range order (LRO) within a parti-

cular phase (iii) local variations in LRO (i.e. modulations) (iv) density

and distribution of ordered phases (v) type, density and distribution of

order-domain boundaries.  The primary techniques of investigation were

x-ray diffraction transmission electron microscopy and field-ion microscopy.

Much of the work was performed on <u>neostructural</u> alloys which change

crystal systems and undergo lattice parameter changes as a function of the

degree of order (e.g. $Ni_4Mo$ and CuPt).  It was found that the accompanying

strains played a very important role not only in the kinetic and mechanistic

behavior of the order transformation but also in the resulting mechanical

strength.  In both $Ni_4Mo$ and CuPt isothermal ordering was found to produce

duplex microstructures consisting of a homogeneous component within the

grains and a heterogeneous component at the grain boundaries, both of which

contribute to the transformation in varying amounts depending on the temper-

ature.  The homogeneous component was found to consist of fine, contiguous,

partially ordered domains which could be viewed as the product of either

fine-scale nucleation and growth or continuous ordering. Kinetic studies showed that this transformation can be described in terms of a continuous ordering model when both strain and order modulations are considered. Stress ordering experiments further emphasized the significance of ordering strains by showing that pre-loads could influence the preferential formation and growth of certain variants (domains). This portion of the work clearly shows the transformation product in some systems cannot be accounted for by simple "quasi-chemical" ordering models.

The long-standing question of the "micro-domain" model for short range order was investigated by means of three-dimensional x-ray diffuse scattering measurements. This technique permitted more accurate separation and analysis of local order and size effect parameters. It was found that SRO in this system was best described in terms of rod-like features of Mo atoms aligned in <100> directions. While this configuration bears some similarity to that of fully-ordered $Ni_4Mo$ it does not represent a clearly-defined picture of "ordered islands". This indicates that the micro-domain model is an over-simplified concept.

The mechanical properties of neostructural ordered alloys was found to be very much dependent on lattice strain and interface strain at order twin boundaries. A direct relationship was found between yield strength strain measured by x-ray line profile analysis. The geometric constraints of lattice periodicity also affect the mechanical properties as expected through their influence on the available shear systems. Deformation twinning was found to occur with relative ease in CuPt although this is generally regarded as an unlikely deformation mode in order structures. Analysis of atomic configurations shows that the usual partial twinning shears may operate in certain variants and preserve the ordered structure while re-orienting the

lattice. The net result is that the geometric restrictions on slip imposed by ordering may, in fact, enhance twinning.

Recent work (nearing completion) on two-phase non-stoichiometric $Ni_4Mo$ alloys deal with the mechanical properties of age-hardening via ordered precipitates. Variations in flow stress with temperature indicate that misfit strain is again an important contribution to the overall strength. In the microstructure range where particle shearing occurs, APB energy also contributes.

The preceding paragraphs summarize most important contributions of the program to the state of knowledge of the microstructure and mechanical behavior of ordered alloys. The work dealt for the most part with room temperature tensile behavior of stoichiometric alloys. It is suggested that this type of work be extended to include the following: (a) non-stoichiometric alloys of varying volume fractions, particle sizes and temperatures, (b) cyclic loading conditions and (c) elevated temperatures, (d) ternary alloying additions.

In addition to the above contributions of the program to the state of knowledge in the field, subsidiary developments of techniques and analysis also evolved during the course of the work. One of these was an analytical technique for calculating the LRO parameter from x-ray data for alloys of preferred domain orientation (LeFevre and Starke, Advances in X-ray Analysis, vol. 12) was used in the order kinetics study. Another was the use of field-ion microscopy techniques in the study of domain sizes beyond the resolution capability of transmission electron microscopy. A review on the past use and potential of this technique in the study of ordered alloys was published during this work.

II.  Publications

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