AD-A129 782 LAVES PHASE FORMATION AND HARDENING OF MARTENSITIC 1/1
FE-9NI-9CO-TI ALLOYS(U) ARMY MATERIALS AND MECHANICS
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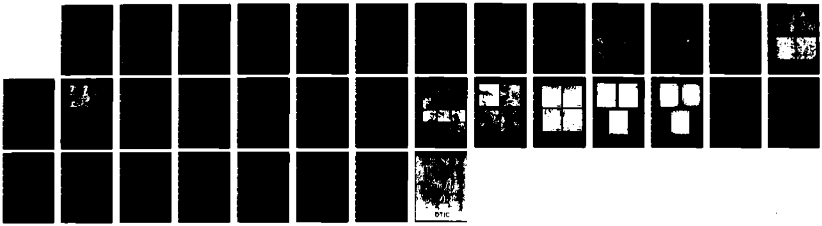

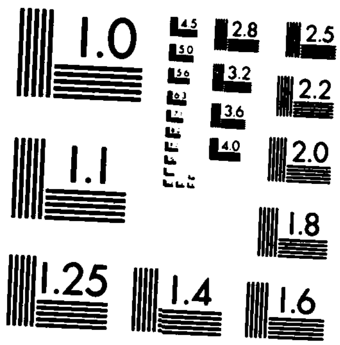

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LAVES PHASE FORMATION AND HARDENING OF Fe-9Ni-9Co-Ti ALLOYS

WEILY F. CHIAO
METALS RESEARCH DIVISION

May 1983

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER AMMRC TR 83-19	2. GOVT ACCESSION NO. AD-A129 782	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) LAVES PHASE FORMATION AND HARDENING OF MARTENSITIC Fe-9Ni-9Co-Ti ALLOYS		5. TYPE OF REPORT & PERIOD COVERED Final Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Weily F. Chiao		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Army Materials and Mechanics Research Center Watertown, Massachusetts 02172		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: 1T0611091A AMCMS Code: 611101-01-A84
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Materiel Development and Readiness Command, Alexandria, Virginia 22333		12. REPORT DATE May 1983
		13. NUMBER OF PAGES 27
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Martensite
Phase transformation
Titanium alloys
Metallography
Age hardening
Iron alloys
Cobalt alloys
Nickel alloys
Laves phase formation

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

A new alloy series, Fe-9%Ni-9%Co with 0 to 5% Ti, has been designed to investigate the formation and hardening of Laves phases in Fe-base alloys. The general microstructural features and hardness properties were investigated after different heat treatments. In addition, the age hardening process was studied. Laves precipitates were formed in the forged, normalized, and annealed conditions. The age hardening was due to the precipitation of Laves phases from the solution-treated matrix. Maximum hardening was developed by aging the solution-treated 5%-Ti alloy at 500°C for 10 hr. No hardening response was observed in the Ti-free alloy. Both optical and scanning electron microscopy were used to follow the aging process and the morphological changes of the precipitate phases. Analysis of precipitation kinetics and morphology indicates that the nucleation of Laves phases is controlled by the martensitic substructure, particularly the martensitic lath boundaries. The level of hardening obtained is significantly greater than that reported for similar ferritic and austenitic steels. Identification of the Laves phases chemistry by microprobe analysis showed the precipitates in the matrix proper to be the Fe_2Ti type compound while the precipitates along grain boundaries are the Ti_4Fe_2O and/or Ti_2Ni type compounds; all precipitates were in the complex forms with possible substitutions among the Fe, Ni, and Co atoms, $(Fe, Ni, Co)_2$ Ti and Ti_2 (Fe, Ni, Co).

CONTENTS

INTRODUCTION

In Fe-base alloys, precipitation hardening by intermetallic phases plays a key role in the development of new materials with optimum mechanical properties.[1,2] Among the various types of intermetallic compounds that can be used as the strengthening agents, the Laves phases have been the least studied. Laves phases are intermetallic compounds of the general formula AB_2. Since the original work by Laves and White[3] in Mg binary and ternary systems, the formation of Laves phases among transition elements has also been extensively studied.[4,5] All investigations were limited to the determination of lattice parameters and physical properties of various binary Laves phases. Little has been done on how to incorporate these phases into new alloy systems to produce materials with high strength and hardness. The objective of this investigation is to develop optimum hardness properties from the formation of Laves phases in a martensitic Fe-9Ni-9Co base alloy with Ti as the additional element for Laves phase formation. The purpose of utilizing Fe-9Ni-9Co as the base alloy was to produce a martensitic ferrite matrix (from Co) solid solution strengthened (from Ni) in which precipitation of Laves phases (Fe_2Ti and/or Co_2Ti) can produce the best hardening properties.

MATERIALS AND PROCEDURES

Four initial heats (25 lb each) were made in a vacuum induction furnace. The melt was poured under an atmosphere of 200-mm Ar at a temperature of approximately 1540°C. A tapered cast iron mold was used for casting in order to avoid the formation of center line shrinkage. All the ingots were hot pressed into 5/8 in. square bars with an intermediate reheat at 1150°C for 30 min. The actual chemical analysis and hardness values of the forged specimens are listed in Table 1. The first ingot (H858), the matrix alloy, contains no Ti. The other three ingots (H893, H894, H895) contain nominally 1, 2.5, and 5% Ti. The contents of interstitial elements are low and uniform for all the alloys.

The central tasks of this investigation were to verify that Laves phases could be formed by the presence of Ti in the Fe-9Ni-9Co base alloy with different heat treatments and that the precipitation of Laves compounds would produce a significant age hardening reaction. Since the alloy series represents a new system, no data of phase relations and T-T-T (time-temperature-transformation) diagrams are available in the literature. This lack of information was remedied by making two preliminary determinations:

1. LENA, A. J. Precipitation Reactions in Iron-Base Alloys, in *Precipitation From Solid Solution,* ASM, Cleveland, Ohio, 1959, p. 244.
2. DECKER, R.F., and FLOREEN, S. Precipitation From Substitutional Iron-Base Austenite and Martensite Solid Solution, in *Precipitation in Iron-Base Alloys,* Gordon and Breach Science Publishers, 1963, p. 69.
3. LAVES, F., and WHITE, H. *Die Kristallstruktm der $MgNi_2$ and seine Bejiehunger Zu den Typen des $MgCu_2$ and $MgZn_2$.* Metallwirtschaft, v. 14, 1935, p. 645.
4. KUO, K. *Ternary Laves and Sigma-Phases of Transition Metals,* Acta Met., v. 1, 1953, p. 270.
5. ELLIOT, R. P., and ROSTOKER, W. *The Occurrence of Laves-Type Phase Among Transition Elements,* Trans. ASM, v. 50, 1957, p. 617.

Table 1. CHEMICAL COMPOSITION AND HARDNESS OF FORGED Fe-9Ni-9Co-Ti ALLOYS

Heat No.	Composition (WT%) Ni	Co	Ti	C	O	N	Hardness DPH (30-kg load)
H858	9.11	9.16	--	0.015	0.012	0.002	199.4
H893	8.88	9.33	0.94	0.015	0.022	0.002	293.0
H894	8.92	9.19	2.32	0.015	0.038	0.001	358.5
H895	8.95	9.73	4.80	0.020	0.011	0.002	428.3

1. the solid solubility of Ti in the Fe-9Ni-9Co base alloy and,

2. the A_s and M_s temperatures in the austenite $\rightleftarrows$ martensite transformation of this new alloy system.

From these determinations, the necessary information for understanding Laves phase hardening can be achieved in terms of formation of martensite, effects of Ti, identification of Laves phases, and the kinetics of precipitation hardening. The experimental procedures of this investigation consisted of the following steps:

1. Investigation of the general microstructural features and hardness of the forged, normalized, annealed, and solution-treated conditions,

2. Determination of solid solubility of Ti in the base alloy by lattice parameter and electrical resistance measurements,

3. Study of the austenite $\rightleftarrows$ martensite transformation during the heating-cooling cycle with electrical resistance vs temperature plots,

4. Characterization of age hardening behavior by microstructural examination and hardness measurements,

5. Identification of Laves phases by electroprobe analysis.

Based on these results, alloy compositions, microstructures, and thermal treatments can be developed for maximum hardening by Laves phase precipitation. Hardening mechanisms can be correlated with Laves phase formation, growth, and morphology.

EXPERIMENTAL RESULTS

Microstructure and Hardness of the Forged, Normalized, Annealed, and Solution-Treated Conditions

A normal experimental procedure of precipitation hardening was established by studying the metallographic features and hardness properties of the four alloys in the forged, normalized (air-cooled), annealed (furnace-cooled), and solution-treated (quenched) conditions. Twelve specimens were prepared from each forged alloy for each heat treatment at three different temperatures for four different time intervals.

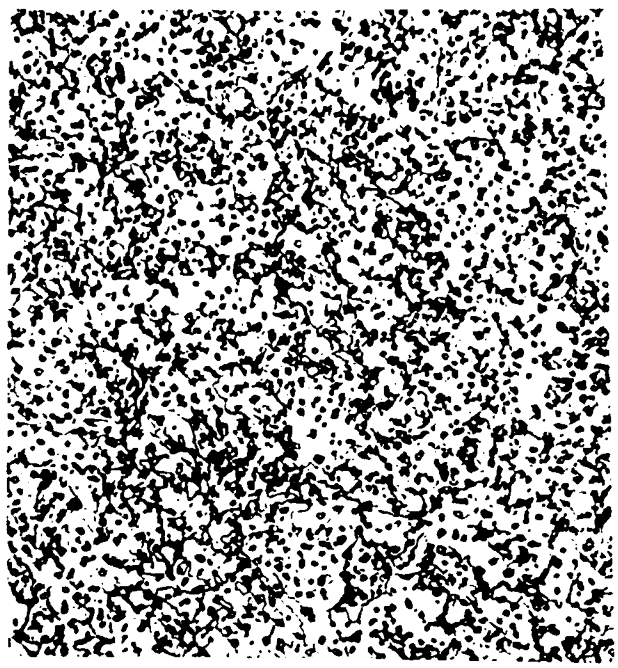
The microstructures of the forged alloys are shown in Figure 1. The base alloy [Figure 1(a)] generally shows polygonal grains without precipitates and the Ti-containing alloys [Figure 1(b)-1(d)] exhibit the fine precipitates uniformly distributed throughout the matrix. Each alloy was then given a normalizing heat treatment at three temperatures; 1000, 1150, and 1300°C for time intervals from 1 to 4 hr. It was found that the microstructure and hardness are mainly related to the Ti content and normalizing temperature. Figure 2 shows the microstructure of the alloys normalized at 1300°C. It can be seen that both the matrix- and Ti-containing alloys display mixed microstructures of ferrite and martensite and that the 5%-Ti alloy [Figure 2(d)] shows grain-boundary melting at 1300°C. Figure 3 shows the hardness response to the normalizing heat treatment. Based on these results, one appropriate normalizing treatment was chosen at 1250°C for 2 hr. to bring all the precipitates of the forged alloys into solution.

Similar studies of annealing heat treatment were also carried out for each alloy composition in the temperature range from 1000 to 1300°C. Higher hardness values were produced from higher annealing temperatures for time intervals up to 2 hr. With longer holding times up to 4 hr., the hardness decreases slightly. The transformation products and their microstructures produced on annealing are quite different from those on normalizing as shown in Figure 4. Fine precipitates exist in the Ti-containing alloys, and the presence of retained austenite becomes quite significant in the 2.5 and 5% Ti compositions.

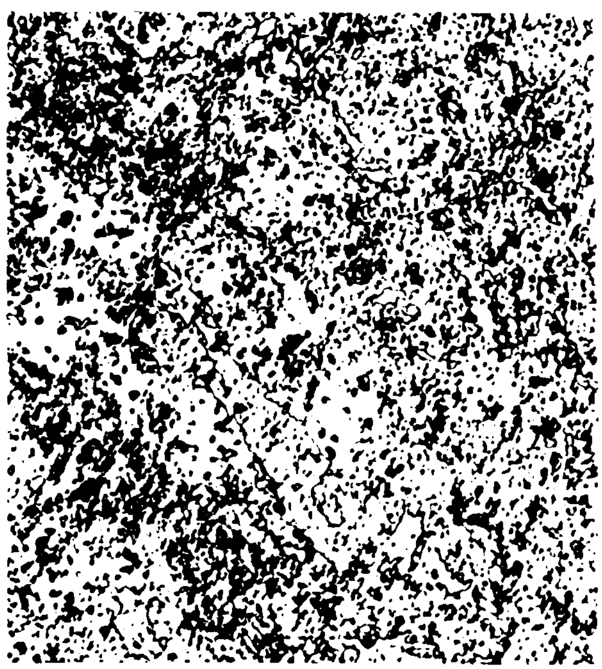
The evaluation of solution treatment has also been done for each alloy at three temperatures; 1000, 1150, and 1300°C. For each solution temperature three different quenching media were applied, e.g., ice brine, water, and oil. It was found that the hardness values and martensite morphology of solution-treated alloys are determined by the Ti content and solution temperatures, but they are not sensitive to the quenching rates obtained from the different quenching media. Therefore, water quenching was used throughout this investigation. The plot of hardness vs solution temperature for different Ti contents is shown in Figure 5(a). Figure 5(b) shows the plot of hardness vs Ti content for different solution temperatures. Based on these preliminary studies and the following determination of solid solubilities, it is considered that 1250°C is the most suitable temperature for bringing the substitutional element Ti into solution. This is verified by the microstructures shown in Figure 6. This figure clearly depicts that the microstructures of solution-treated alloys are all massive or lath martensite, but the details of the martensite morphology of the matrix alloy [Figure 6(a)] are quite different from those of the Ti-containing alloys [Figure 6(b)-6(d)]; the former consists of martensite blocks much finer and thinner than the latter.

In summary, hardness is plotted in Figure 7 as a function of Ti content for all heat treatments under similar solution-treatment conditions (1250°C for 2 hr). It is interesting to note that the order of hardening in the base alloy is just the reverse of that in the Ti-containing alloys. For the Ti-containing alloys the quenched condition yields the lowest hardness due to the absence of precipitates found in the forged, normalized, and annealed conditions. However, for the base alloy the solution-treated condition actually yields, among the different heat-treatments, the highest hardness due to the difference in martensite morphology.

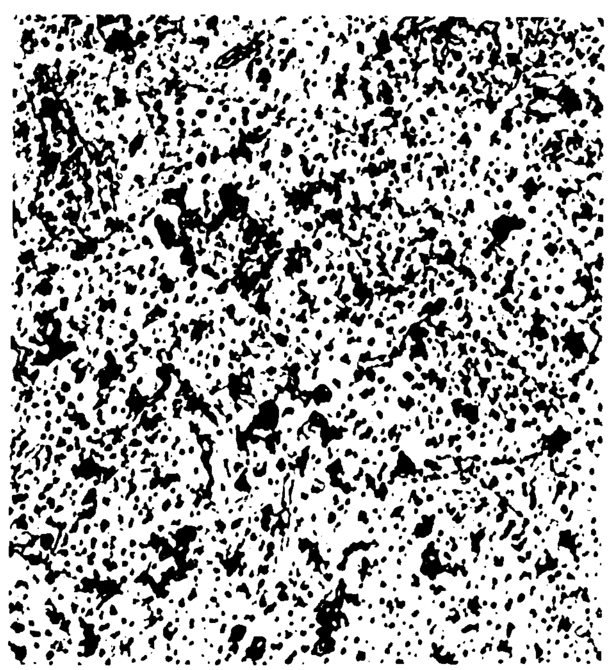
(a.)0% Ti



(b.)1% Ti

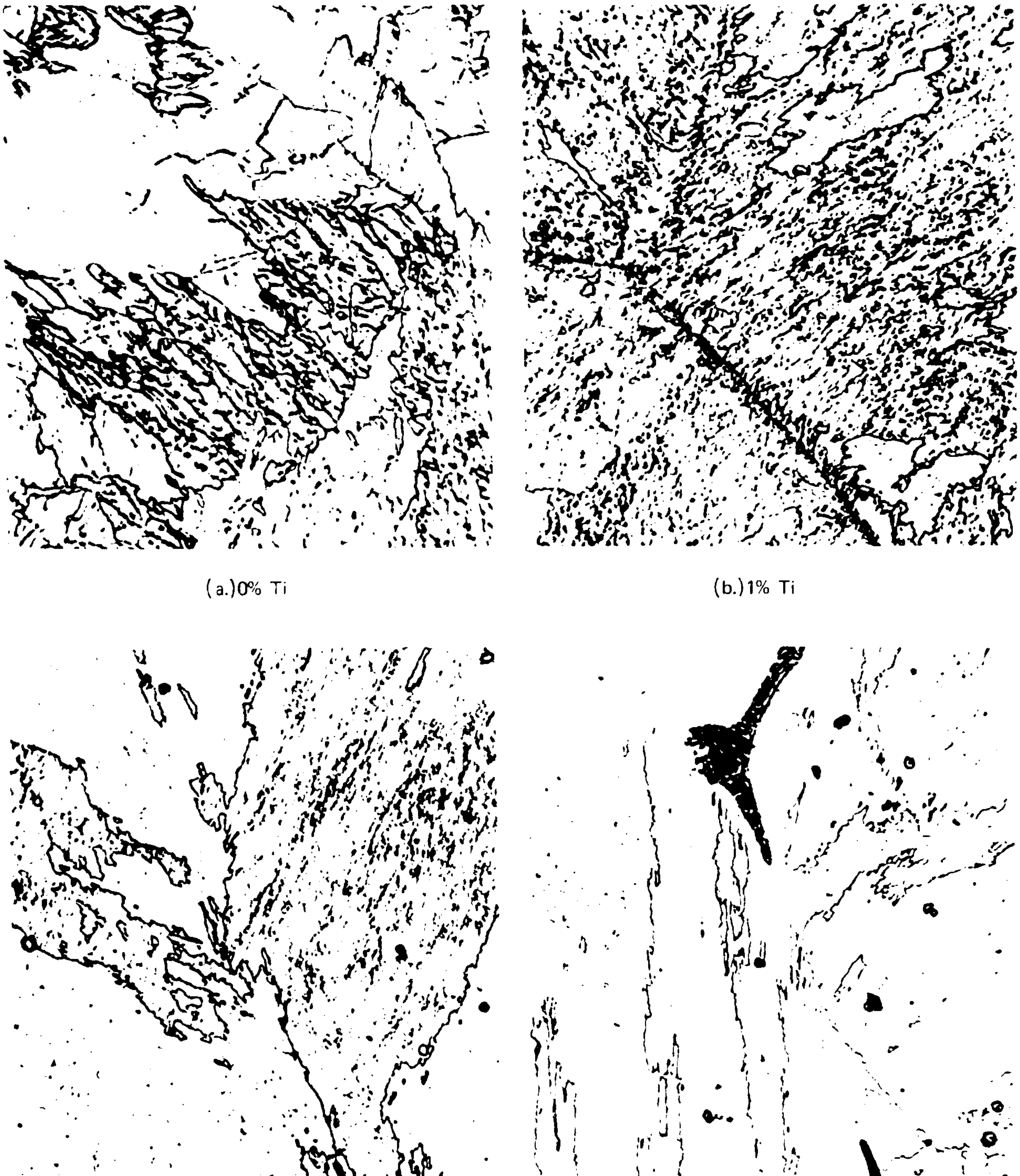


(c.)2.5% Ti



(d.)5% Ti

Figure 1. Microstructures of forged Fe-9Ni-9Co-Ti alloys. Mag. 500X. (Forged at 1150°C with intermediate reheat at 1150°C for ½ hr.)



(a.)0% Ti

(b.)1% Ti

(c.) 2.5 Ti

(d.)5% Ti

Figure 2. Microstructures of normalized Fe-9Ni-9Co-Ti alloys. Mag. 500X. (1300^{o}C/2 hr and air-cooled.)

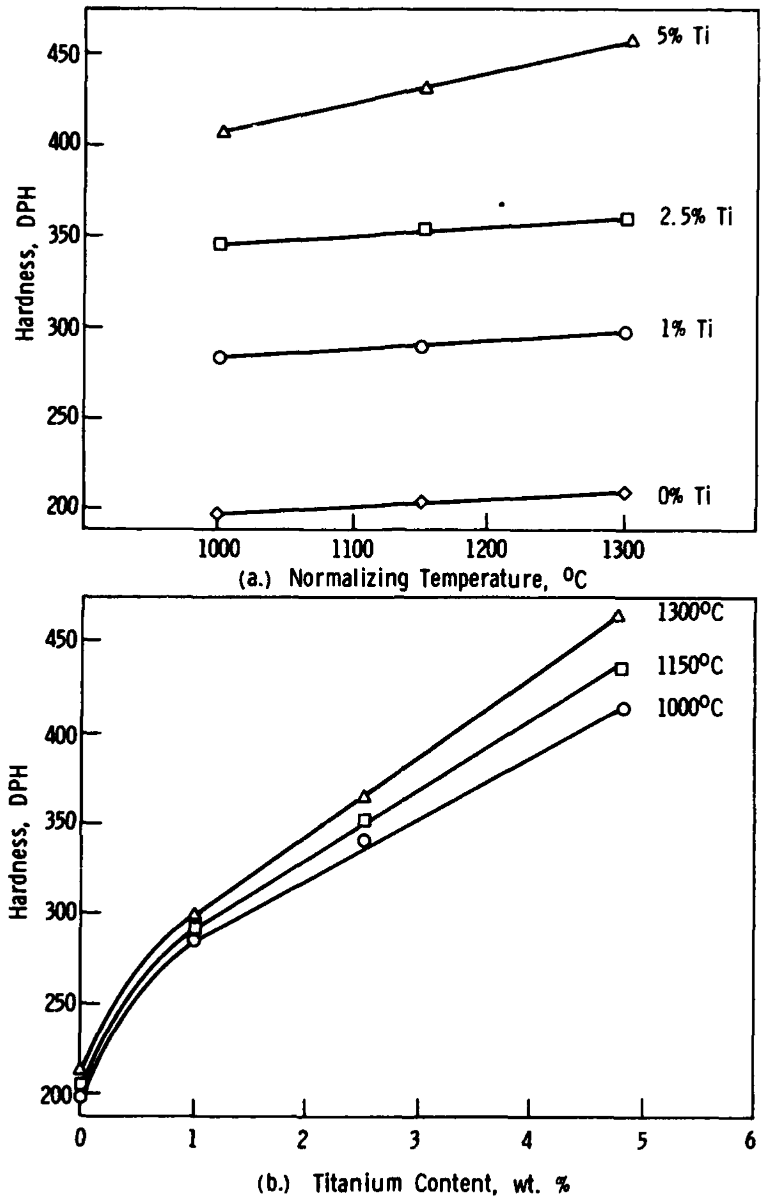


Figure 3. Hardness properties of Fe-9Ni-9Co-Ti alloys normalized at different temperatures.

Solid Solubility of Ti in Fe-9Ni-9Co Matrix

It is well known that Ni and Co exhibit an extended range of solid solution in fcc or bcc iron, but Ti has only limited solid solubility, which decreases with decreasing temperature.[6] For this work, it is necessary to know the change of lattice parameter of Fe due to the presence of 9% Ni and 9% Co in the matrix and

6. HANSEN, M. *Constitution of Binary Alloys*, McGraw-Hill Book Co., New York, 1958.

(a.) 0% Ti

(b.) 1% Ti

(c.) 2.5% Ti

(d.) 5% Ti

Figure 4. Microstructures of annealed Fe-9Ni-9Co-Ti alloys. Mag. 500X. (1300°C/2 hr and furnace-cooled.)

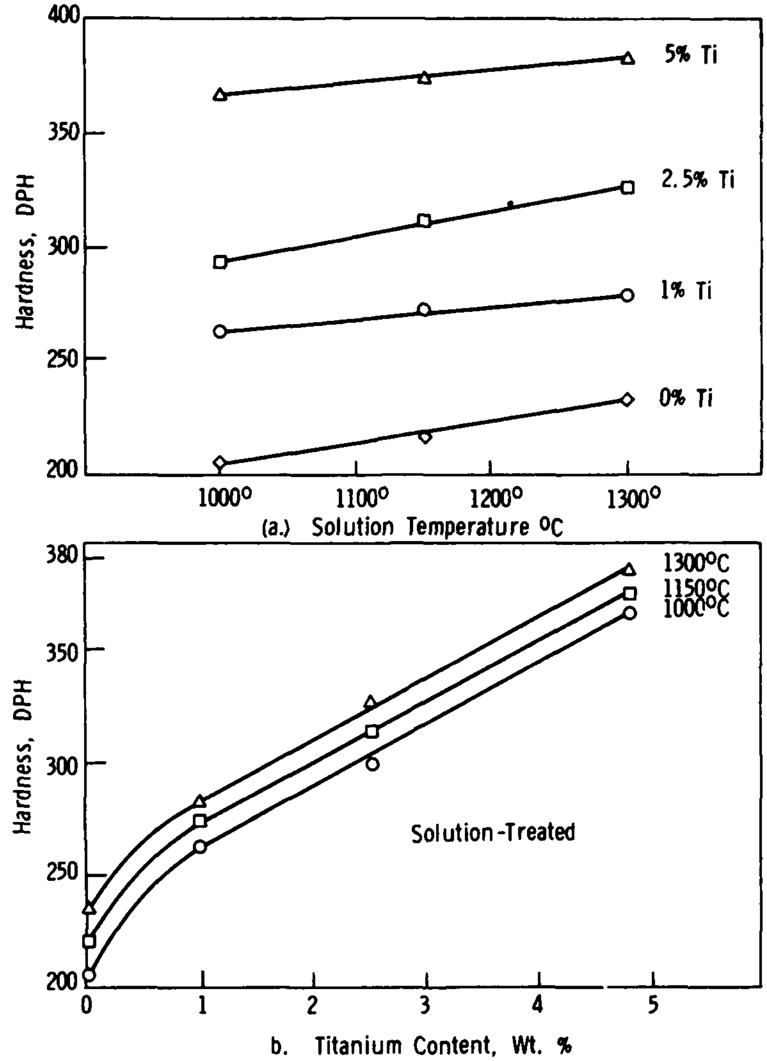
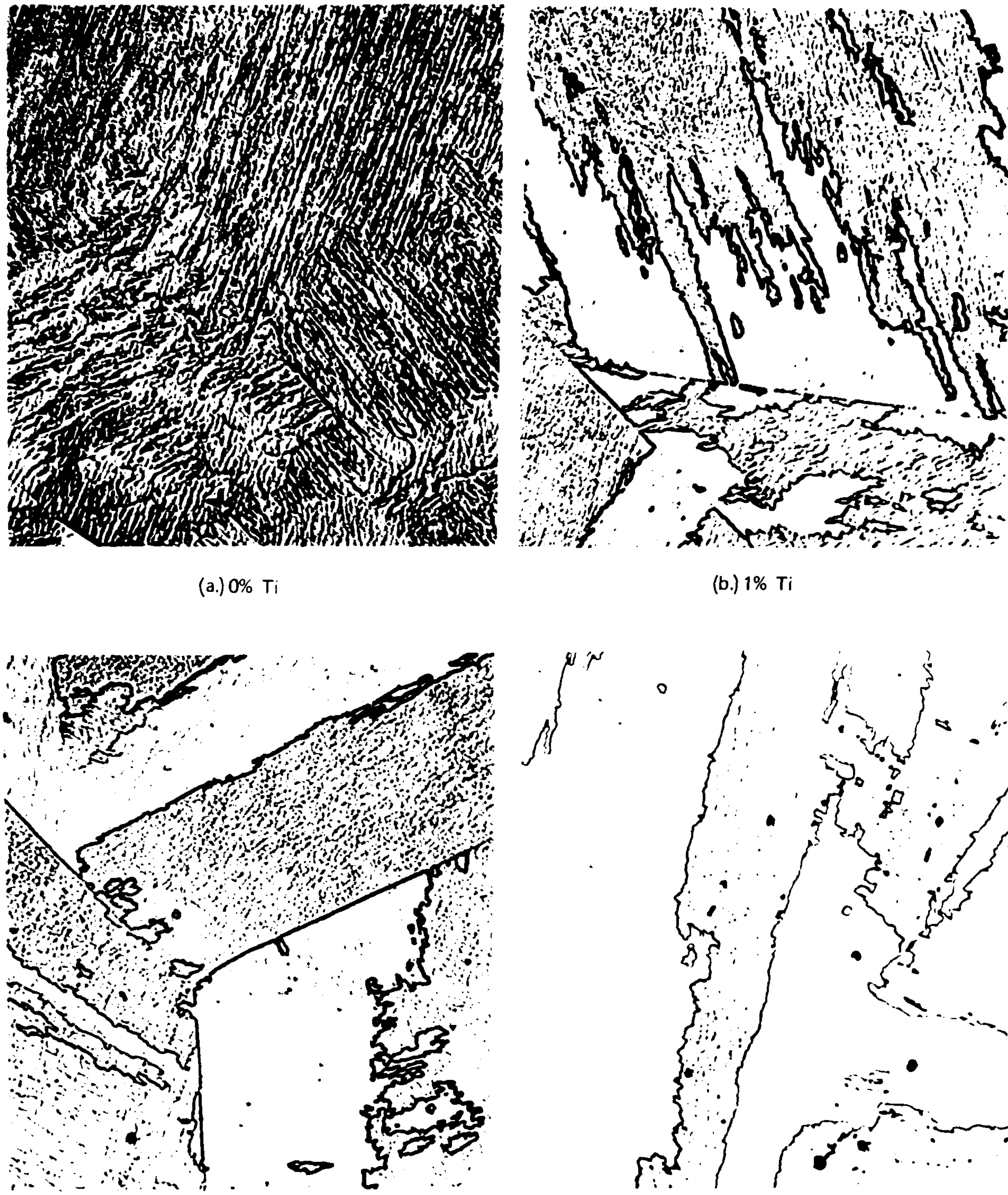


Figure 5. Hardness properties of Fe-9Ni-9CO-Ti alloys solution-treated from different temperatures.

the additions of 1 to 5% Ti. Also, it is necessary to know the solid solubility of Ti in the base alloy at different solution temperatures. The former question utilized the alloy series in the quenched condition; the latter used the 5% Ti alloy quenched from different solution temperatures. Both determinations were done by two parametric methods: lattice parameter and electrical resistance. Both methods yielded the same results that are given in Figure 8. Solubility limits are indicated by the compositions at which the lattice parameter and resistance values flatten.

The solid solubility of Ti in the basic alloy was determined in the temperature range from 1000 to 1300°C in order to select suitable temperatures for solution treatment. Since the precipitation-hardening behavior is related to the solute



(a.) 0% Ti

(b.) 1% Ti

(c.) 2.5 % Ti

(d.) 5% Ti

Figure 6. Microstructures of solution-treated Fe-9Ni-9Co-Ti alloys. Mag. 500X. (1250°C/2 hr and water-quenched.)

content, it is essential to bring the highest Ti content, 5.55% at (4.80% wt) into solution by heat treatment. The present results show that this is accomplished by 1250°C.

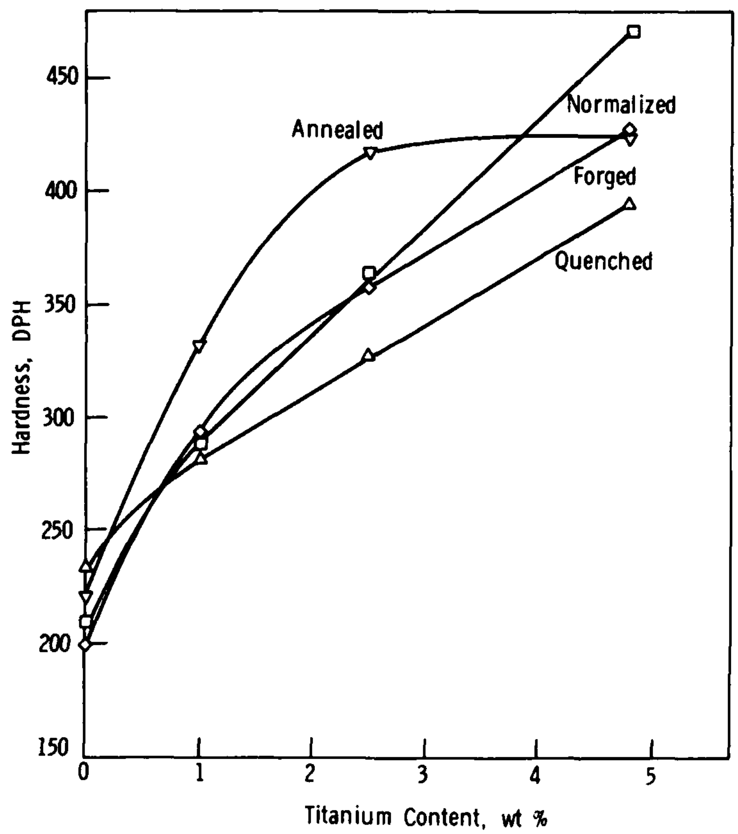


Figure 7. Hardness properties of Fe-9Ni-8Co-Ti alloys after different heat-treatments.

The Martensite ⇄ Austenite Transformation

In order to understand the hardening behavior of Fe-base alloys, one of the most indispensable considerations is the investigation of the martensite (α) ⇄ austenite (γ) transformation from which the A_s and M_s temperatures can be determined. The α ⇄ γ transformation of the Fe-9Ni-9Co-Ti system was followed by electrical resistance measurements at temperatures up to 1000°C with a heating and cooling rate of about 1 to 2°C per minute. From the resulting resistance vs temperature curves, the A_s and M_s temperatures are identified by abrupt changes in resistance on heating and cooling, respectively. The numerical values of A_s and M_s thus determined for the alloy series are listed in Table 2.

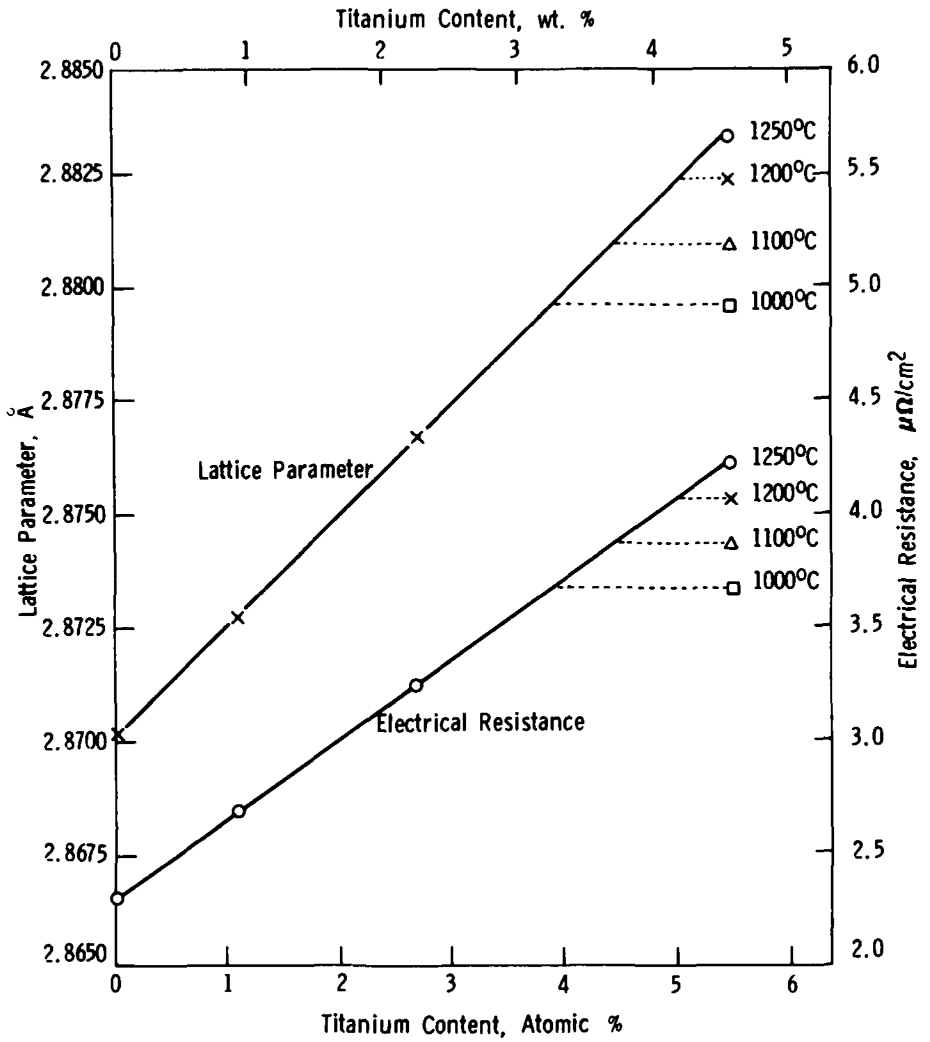


Figure 8. Determination of solid solubility of Ti in Fe-9Ni-9Co matrix.

Table 2. A_s AND M_s TEMPERATURES OF Fe-9Ni-9Co-Ti ALLOYS

Alloy	A_s, °C	M_s, °C
Fe-9Ni-9Co	759	659
Fe-9Ni-9Co-1Ti	763	638
Fe-9Ni-9Co-2.5Ti	793	544
Fe-9Ni-9Co-5Ti	797	544

Prior work on Fe-9%Ni alloy has indicated the A_s at 700°C and M_s at 500°C.[7,8] The present data indicates that the presence of 9%Co in the Fe-9%Ni alloy raises A_s from 700 to 759°C and M_s from 500 to 659°C. This verifies that Co functions as a stabilizer of α-ferrite in the present alloy system. This effect was also observed in alloys of higher Ni-content.[9-11] The present study shows that the Ti additions cause a slight increase of A_s and a large uniform decrease of M_s with respect to the base alloy (Table 2). A similar phenomenon was also observed in Yeo's work[12] on the addition of Ti to the Fe-22.5%Ni alloys. In that work, the rate of M_s decrease is about 22°C per 1% Ti as compared to the 32°C per 1% Ti in this study. Since Ti generally functions as a stabilizer of α-ferrite, it is unclear what effect aging will have on the matrix. From the data presented in Table 2, it seems certain that the present alloy compositions under study would produce a martensitic ferrite matrix with little formation of austenite during aging below 700°C. With Ti as the key element for Laves phase formation, a unique precipitation hardening would be expected on aging.

Characteristics of Age Hardening

The aging behavior of the Fe-9Ni-9Co-Ti system was studied with samples vacuum-annealed at 1250°C for 2 hr, water-quenched and aged in salt baths. The changes in hardness as a function of aging time (from 1 min to 88 hr) during isothermal aging at 500, 600, 700, and 800°C are shown in Figure 9. Peak hardness occurs for all alloy compositions at an aging temperature of 500°C [Figure 9(a)]. Some scattered data at 400°C showed hardness peaks slightly lower than those at 500°C. Maximum hardness decreases with increasing aging temperature [600°C-Figure 9(b), 700°C-Figure 9(c)]. At 800°C, over-aging, not hardening, occurs [Figure 9(d)]. The maximum hardness peaks are generally shifted to shorter times with higher temperatures and increased Ti content [Figures 9(b) and 9(c)]. The base alloy exhibits little change in hardness over the temperature range 500 to 700°C and decreases slightly at 800°C. This is beneficial as it implies that a stable matrix is preserved while high strength is developed by precipitation of the Laves compounds. The initial age-hardening response, i.e., the initial rate of hardness increase, became faster and steeper with either increasing temperature from 500 to 700°C at constant Ti content or increasing Ti content from 1 to 5% at constant temperature [Figures 9(a)-9(c)]. No incubation period appears, as shown by the acceleration of the precipitation reaction at the first minute of aging at 700°C. The over-aging process is slow at 500°C and only becomes predominant as the temperature is increased to 800°C.

7. RUDMAN, P. S. *The Atomic Volumes of the Metallic Elements,* Trans. Met. Soc. AIME, v. 233, 1965, p. 864.
8. TOFANTI, W., and BUTTINGHANS, A. *Arch Eisenhuettenw,* v. 12, 1938-1939, p. 33.
9. COLLING, D. A. *Martensite-to-Austenite Reverse Transformation in Fe-Ni-Co Alloys,* Met. Trans., v. 1, 1970, p. 1677.
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11. KAUFMAN, L., and COHEN, M. *The Martensitic Transformation in the Fe-Ni System,* Trans. AIME, October 1956, p. 1393.
12. YEO, R. B. G. *The Effect of Some Alloying Elements on the Transformation of Fe-22.5 Pct Ni Alloys,* Trans. AIME, v. 227, 1963, p. 884.

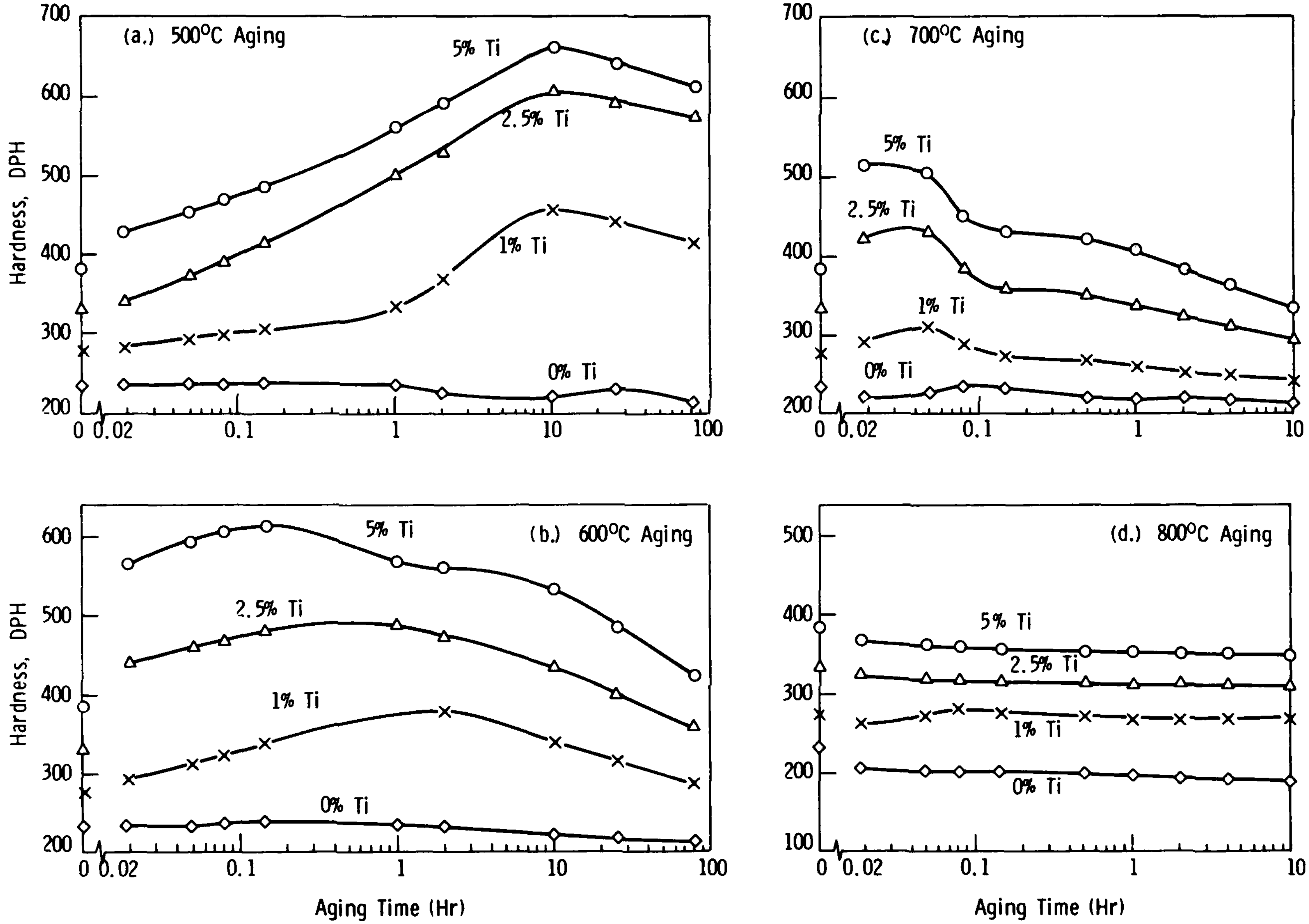


Figure 9. Change of hardness of Fe-9Ni-9Co-Ti alloys at four different aging temperatures.

The microstructures of the alloys aged at 500°C are given in Figure 10. For the base alloy, Fe-9Ni-9Co, [Figures 10(a)-10(d)] no precipitation occurs during the entire period. The essential change in microstructure is the change in morphology of the martensite ferrite. Since no precipitates formed in the base alloy, this indicates that Ti is the key element in forming Laves compounds. For the Ti-containing alloys, precipitation increases with time and Ti-content [Figures 10(e)-10(l)]. All precipitates are finely dispersed. No apparent over-aging appears, even at 88 hr at T = 500°C. Thus, maximum hardness peaks developed at 500°C. As temperatures were increased from 500 to 800°C, over-aging reactions develop. This process is illustrated by the microstructures of the 5%-Ti alloy in Figure 11.

The maximum hardness (670 DPH) for the series was achieved by holding the 5%-Ti alloy at 500°C for 10 hr. The mechanism for this hardening is the dense precipitation of irregularly shaped plates in a closely interlocking arrangement, as illustrated by the scanning electronmicrographs [Figures 12(b)-12(d) and 13(a)]. The over-aging phenomenon appears as the separation of these plates at higher temperatures [Figures 13(b) and 13(c)]. Finally, the increase of the volume fraction of the Laves precipitates with increasing Ti content is shown in the carbon replica photographs in Figure 14.

Identification of Laves Phases

The identification of the Laves phases was done by the electron-probe analysis of the metallographic specimens. Figure 15 shows the second phase precipitates produced by annealing the forged 5%-Ti alloy at 1300°C for 2 hr followed by very slow cooling. The result of electron-probe analysis of the particles in this specimen is summarized in Table 3. These results indicate that the majority of the precipitated phases contain a chemistry similar to the Laves compound Fe_2Ti (30.01 weight% Ti) with some substitution of Fe by Ni and Co. However, the particles precipitated along the grain boundaries appear tan-colored under the polarized microscope, which indicates the presence of oxygen; their chemistry shows a different type of Laves compound Ti_4Fe_2O, also with possible substitution of Fe by Ni and Co. This compound is reported to be similar to the $FeTi_2$ (63.17 weight% Ti) type Laves phases. The formation of these two types of Laves phases will be explained in the discussion.

Table 3. MICROPROBE CHEMISTRIES OF SECOND PHASES IN Fe-9Ni-9Co-5Ti ALLOY (WEIGHT%)

Area	Ti	Fe	Co	Ni	Remarks
Large Particles	34.6	53.5	6.9	5.3	$(Fe\text{-}Co\text{-}Ni)_2Ti$
Small Particles*	24.5	62.7	7.2	5.0	$(Fe\text{-}Co\text{-}Ni)_2Ti$*
Grain Boundary Tan Particles	63.0	28.4	2.7	2.8	Ti_4Fe_2O and/or $Ti_2(Fe,Co,Ni)$

*Includes unavoidable matrix excitation.

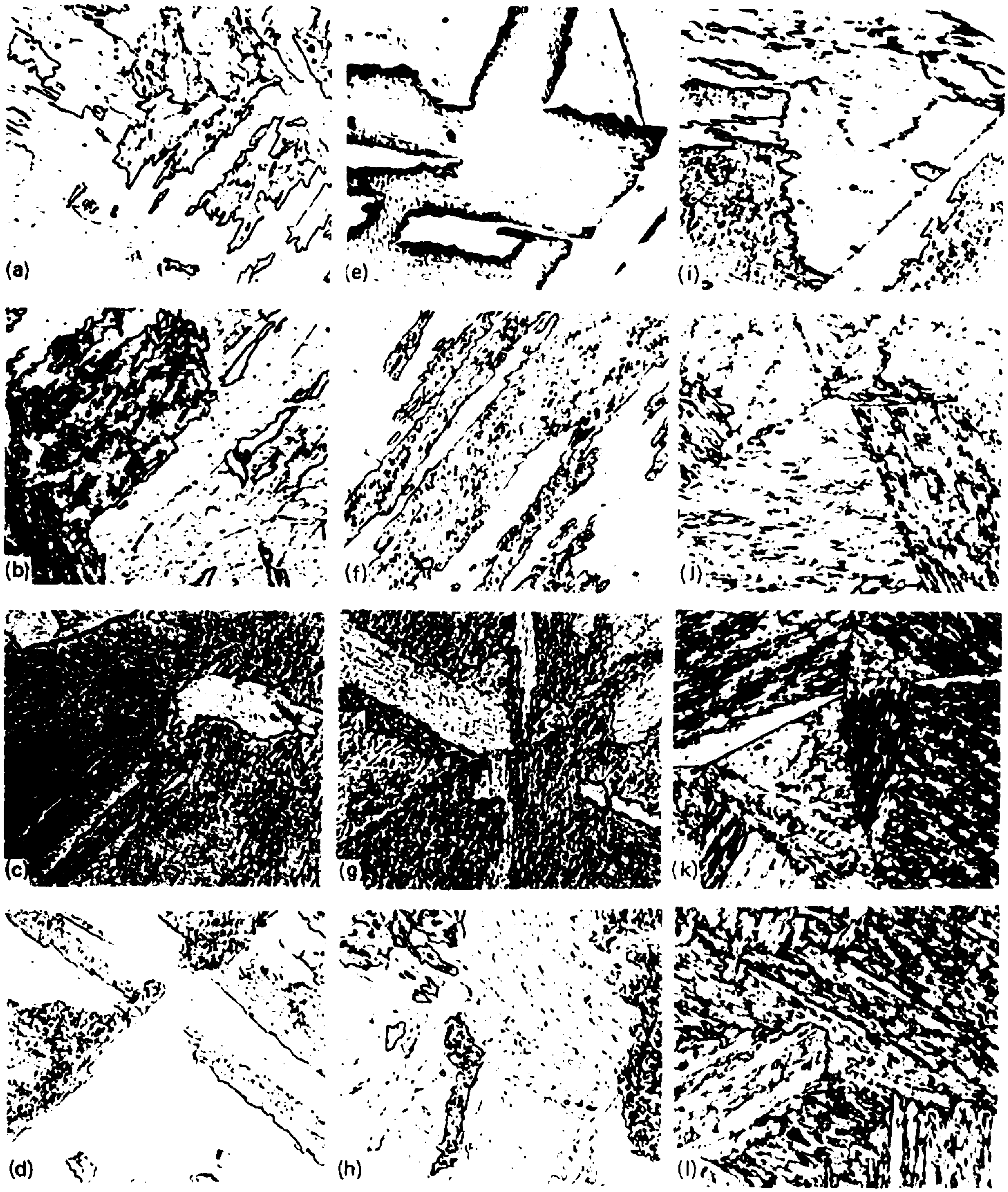
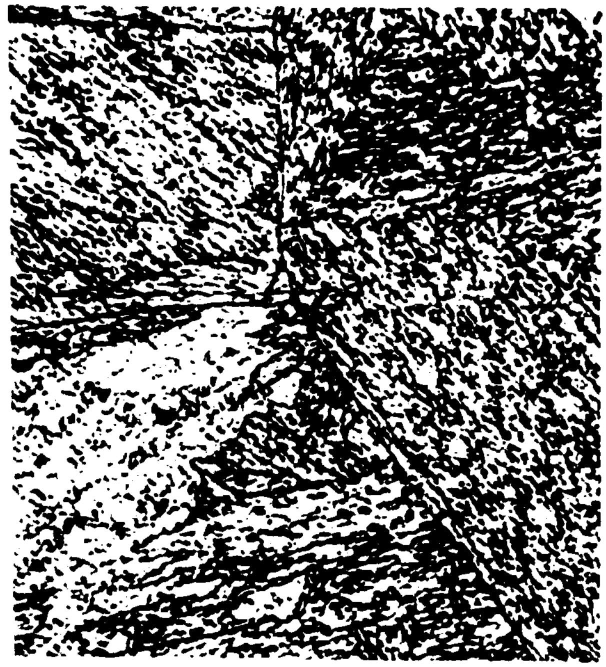


Figure 10. Microstructures of Fe-9Ni-9Co-Ti alloys after aging at 500°C for different time intervals. Mag. 500X. (Solution-treated at 1250°C/2 hr.)

(a.)500°C, 25 hr



(b.)600°C, 84 hr

(c.)700°C, 10 hr

(d.)800°C, 10 hr

Figure 11. Microstructures of Fe-9Ni-9Co-5Ti alloy solution-treated at 1200°C and then aged at various temperatures for different time.

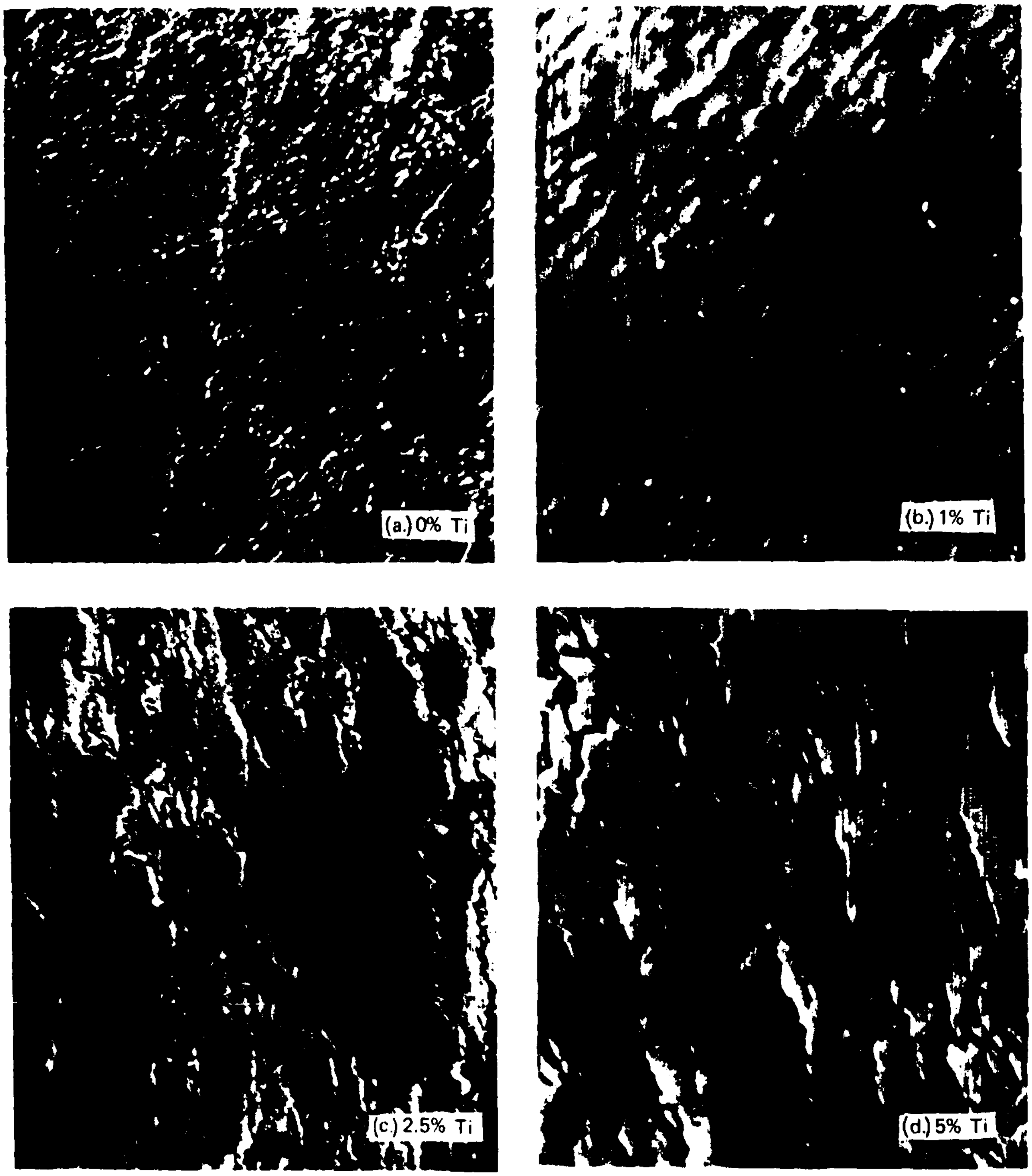
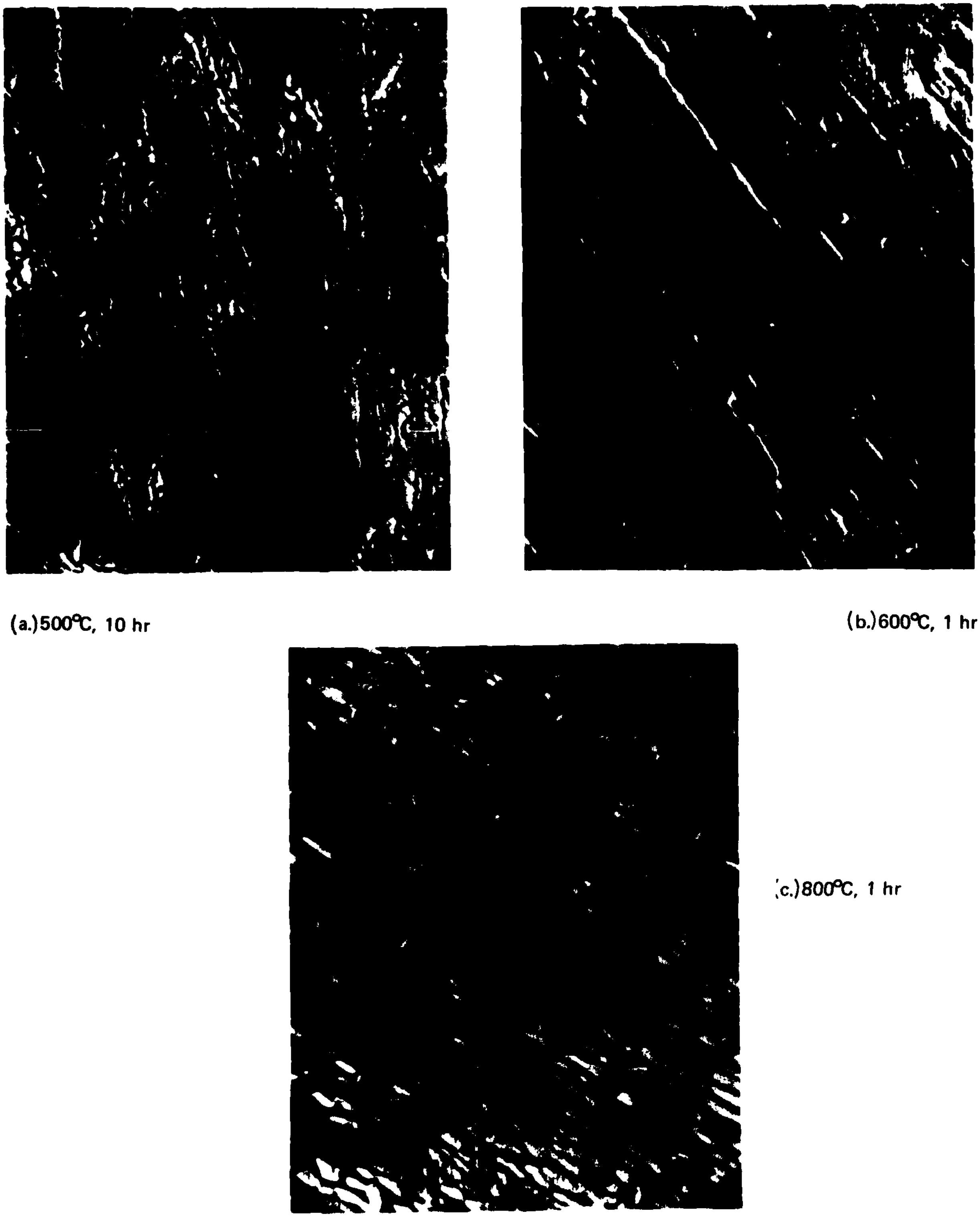


Figure 12. Electron scanning microscopic cross-sections of Fe-9Ni-9Co-Ti alloys aged at 500°C for 25 hr. Mag. 4000X.

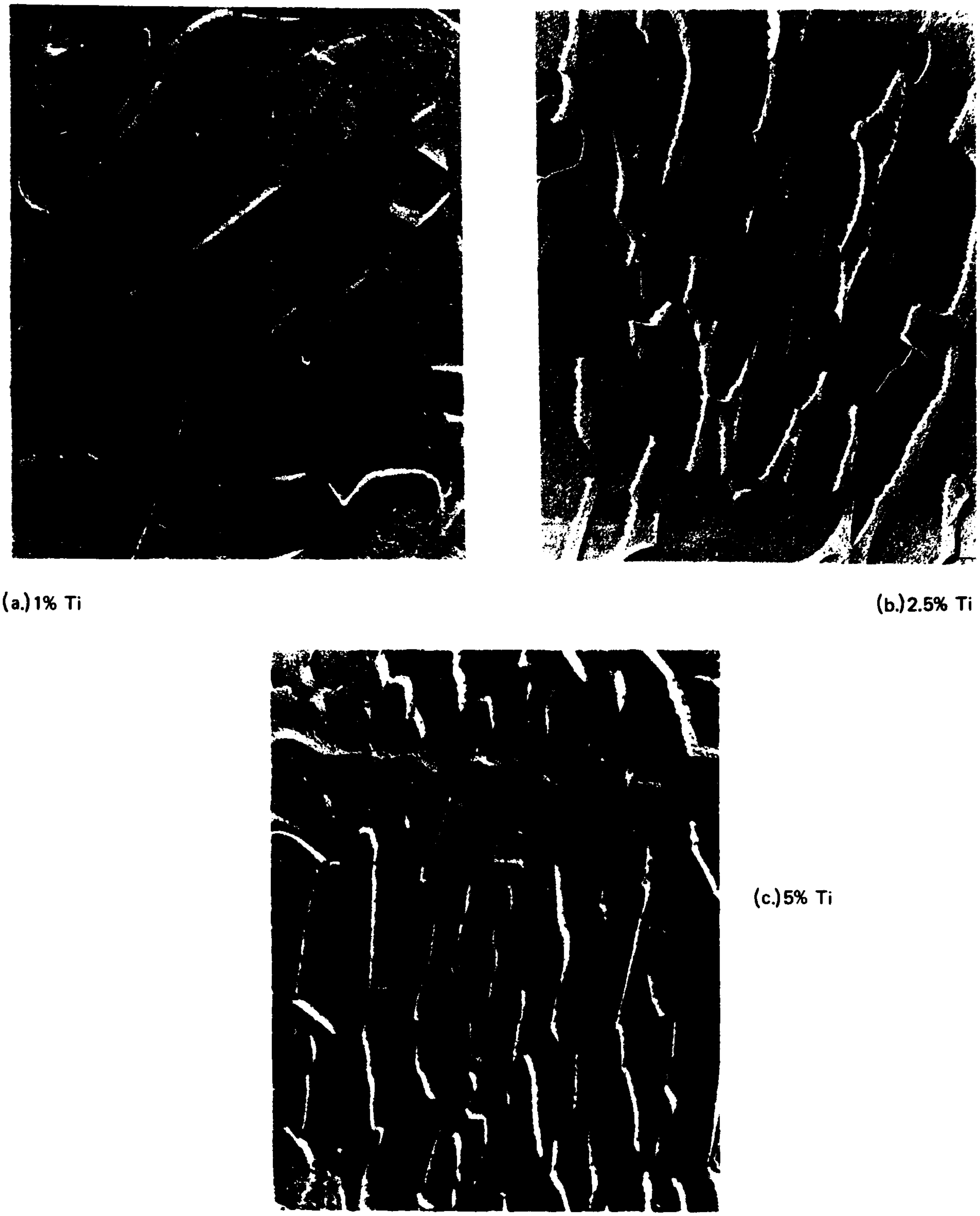


(a.)500°C, 10 hr

(b.)600°C, 1 hr

(c.)800°C, 1 hr

Figure 13. Electron scanning microscopic sections of Fe-9Ni-9Co-5Ti alloy aged at three different temperatures. Mag. 4000X.



(a.) 1% Ti

(b.) 2.5% Ti

(c.) 5% Ti

Figure 14. Electron replica photography showing increasing amounts of precipitates with Ti contents in Fe-9Ni-9Co-Ti alloys aged at 800°C for 17 hr. Mag. 10,000X.

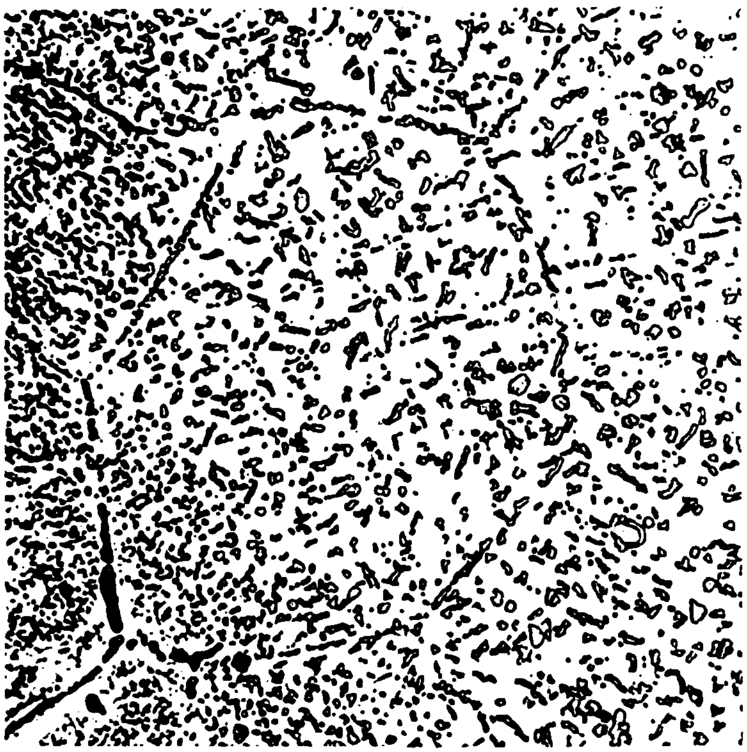


Figure 15. Laves precipitates after annealing at 1300°C for 2 hr followed by very slow cooling of the forged Fe-9Ni-9Co-5Ti alloy. Mag. 500X.

DISCUSSION

The Formation of Laves Phases in Fe-9Ni-9Co-Ti Alloys

One aim of this investigation was to demonstrate that Laves phases could be formed with the addition of the key element Ti to the Fe–9Ni–9Co base alloy with various thermal treatments. The present results show that such Laves phases did occur in Ti-containing alloys in all heat-treated conditions. The most interesting discovery was that two types of Laves phases exist: a Fe_2Ti type compound precipitating in the matrix-proper and Ti_4Fe_2O and/or Ti_2Ni type compound along the grain boundaries. All these types are complex forms with possible substitutions among Fe, Ni, and Co. In studies of formation of intermetallic compounds in the Ti-Fe, Ti-Ni, and Ti-Co systems, there were controversies regarding the existance of Ti_2Fe.[13-16] Recent investigations[17,18] confirmed that Ti_2Fe is not an equilibrium phase. The presence of a very small amount of oxygen can shift the alloy composition into a ternary phase involving the formation of Ti_4Fe_2O whose structure resembles

13. LAVES, F., and WALLBAUM, H. J. *NaturWissenschafteu*, v. 27, 1939, p. 674.
14. DUWEY, P., and TAYLOR, J. L. *The Structure of Intermetallic Phases in Alloys of Ti with Fe, Co and Ni*, Trans. AIME, v. 188, 1950, p. 1173.
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that of Ti_2Fe. As to the other two isomorphous compounds, Ti_2Ni and Ti_2Co, both their similarities in the formation and structure have been well established.[19,20] It is thus concluded that the Laves precipitates along the grain boundaries are Ti_4Fe_2O and Ti_2Ni type compounds (with the possible substitutions among Fe, Ni, and Co).

The formation of several types of Laves phases in this material creates an unusual case. This dual precipitation creates peculiar structures along grain boundaries. The interfacial structures that result exert an influence on the hardening properties. Since the crystallographic configuration along the interfacial areas was not determined, no prediction can be made here on the influence of dual formation of Laves precipitation upon the hardening properties of the experimental alloys. Since it is generally believed that a cubic structure (Ti_2Ni) may be more ductile than the hexagonal structure (Fe_2Ti), the formation of cubic Ti_4Fe_2O type compound in the grain boundary area could be a beneficial phenomenon for the development of optimum mechanical properties.

The Kinetics and Morphology of Laves Phase Precipitation in Martensitic Fe-9Ni-9Co-Ti Alloys

The study of aging characteristics of the present alloy series (Figure 9) has shown that significant hardening can be achieved with Laves phase precipitation. The kinetics of aging, using the general equations of Lement and Cohen,[21] can be expressed as

$$H_t - H_o = Kt^n \quad ,$$

where H_t is the hardness at aging time t, H_o the initial hardness, K a constant, and n the time or growth exponent. Figure 16 illustrates this relationship for the Ti-containing alloys at two temperatures, 500°C (Figure 16) and 600°C [Figure 16(b)]. The values of the time exponent n, the slopes of these plots, varies with the aging temperature, time, and Ti content. In this study, the magnitude of n in the initial stages generally decreases with increasing aging temperature and Ti-content. The value of n ranges from 0.48 to 0.10. Several theoretical studies of the mechanisms of solute precipitations in iron-base alloys exist. Zener[22] showed that the lowest possible value of n would be 0.5 if volume diffusion of solute atoms is the basic mechanism of the growth of spherical precipitates from solid solution. Lament and Cohen[21] studied the first stage of tempering of carbon steels using a dislocation attraction model, and demonstrated that lower n values in the range observed here

19. YURKO, G. A. *The Crystal Structure of Ti_2Ni,* Acta. Cryst., v. 12, 1959, p. 909.
20. ORRELL, F. L., and FONTANA, M. G. *The Titanium-Cobalt System,* Trans. ASM, v. 47, 1955, p. 554.
21. LAMENT, B. S., and COHEN, M. *A Dislocation Attraction Model for the First Stage of Tempering,* Acta Met., v. 4, 1956, p. 469.
22. ZENER, C. *Theory of Growth of Spherical Precipitates from Solid Solution,* J. App. Phys., v. 20, 1949, p. 950.

can be associated with the defect-assisted diffusional growth of heterogeneously nucleated precipitates.

Heterogeneous precipitation on the lath martensitic substructure is strongly suggested by the highly-oriented morphology of precipitation evident in Figures 11 through 14. The precipitate plates appear aligned with the lath packet structure in a manner highly suggestive of precipitation on martensitic lath boundaries (e.g., Figures 12 and 13). If the precipitation kinetics are dominated by the martensitic substructure, a possible benefit of Co in these alloys may be the inhibition of recovery of this substructure as observed in a study of the tempering behavior of 10Ni-8Co steels.[23]

Further support for the importance of the role of martensitic substructure comes from comparison of the level of hardening obtained in this study with those of previous studies of Ti Laves phase hardening in other steels, Table 4. The hardening observed in the current martensitic steels is substantially greater than that obtained in similar ferritic and austenitic steels.

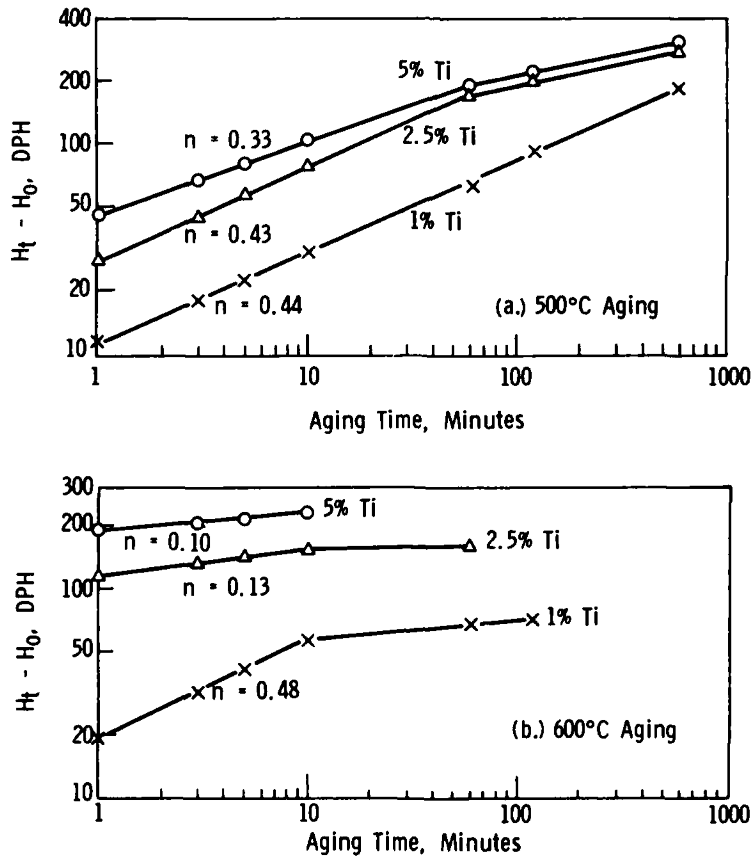

Figure 16. Change in hardness vs aging time at (a) 500°C and (b) 600°C for Fe-9Ni-9Co-Ti alloys.

23. SPEICH, G. R., DABKOWSKI, D. S., and PORTER, L. F., *Met. Trans.*, v. 4, 1973, pp. 303-315.

Table 4. LAVES PHASE HARDENING IN Fe-BASE ALLOYS

Alloy System	Matrix Type	Weight% Ti	Aging Treatment	Max Hardness (DPH)	Reference
Fe-Ti	Ferritic	5.8	700°C, 1 hr	310	G. R. Speich[24]
		8.8	600°C, 10 hr	485	
Fe-9Mi-9Co-Ti	Martensitic	2.3	500°C, 10 hr	610	this work
		4.8	500°C, 10 hr	665	
Fe-15Cr-20Ni-Ti	Austenitic	2.7	700°C, 40 hr	340	R. Blower[25]
		3.8	700°C, 25 hr	400	

Thus, it appears very promising to develop optimum mechanical properties from the present Fe-9Ni-9Co-Ti alloy system due to its much higher hardening properties.

SUMMARY

1. A new alloy series Fe-9%Ni-9%Co (1 to 5% Ti) has been investigated in order to develop optimum hardening properties by the formation of Laves phases.

2. The general features of the microstructures and hardness properties of the alloys were investigated in the forged, normalized, annealed, and solution-treated conditions.

3. The solid solubility of Ti in the Fe-9Ni-9Co matrix was determined as 5.5 at. pct. (4.7 %weight) Ti at 1250°C by lattice parameter and electric resistance measurements.

4. The martensite $\rightleftarrows$ austenite transformations of the whole series were studied with resistance vs temperature plots, from which the A_s and M_s temperatures of each alloy composition were determined. Ti additions lower M_s and slightly raise A_s.

5. No aging response was observed in the matrix alloy. The maximum hardness of 670 DPH was developed by solution treating the 4.8 %-Ti alloy at 1250°C for 2 hr, followed by aging at 500°C for 10 hr.

6. The kinetics and morphology of precipitation indicate control by the martensitic substructure. The resulting level of precipitation hardening is significantly greater than that reported for similar ferritic and austenitic steels.

7. The identification of the Laves phase chemistry by microprobe analysis showed that the precipitate in the matrix proper is the type Fe_2Ti compound and the precipitate along grain boundaries is the Ti_2Ni and/or Ti_4Fe_2O type compound, both in the complex forms with possible substitutions among the Fe, Ni, and Co, $(Fe, Ni, Co)_2Ti$ and $Ti_2(Fe, Ni, Co)$.

24. SPEICH, G. R. *Precipitation of Laves Phases from Fe-Nb and Fe-Ti Solid Solution,* Trans. AIME, v. 224, 1962, p. 850.
25. BEETTIC, H. J., JR., and HAGAL, W. C. *Intermetallic Compounds in Titanium-Hardened Alloys,* Trans. AIME, J. of Metals, July, 1957, p. 911.

ACKNOWLEDGEMENT

The author wishes to thank Drs. Gregory Olson and Judy Kohatsu for their enthusiastic discussions and comments.

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Metallography	Laves phase formation
Age hardening	

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A new alloy series, Fe-9%Ni-9%Co with 0 to 5% Ti, has been designed to investigate the formation and hardening of Laves phases in Fe-base alloys. The general microstructural features and hardness properties were investigated after different heat treatments. In addition, the age hardening process was studied. Laves precipitates were formed in the forged, normalized, and annealed conditions. The age hardening was due to the precipitation of Laves phases from the solution-treated matrix. Maximum hardening was developed by aging the solution-treated 5%-Ti alloy at 500°C for 10 hr. No hardening response was observed in the Ti-free alloy. Both optical and scanning electron microscopy were used to follow the aging process and the morphological changes of the precipitate phases. Analysis of precipitation kinetics and morphology indicates that the nucleation of Laves phases is controlled by the martensitic substructure, particularly the martensitic lath boundaries. The level of hardening obtained is significantly greater than that reported for similar ferritic and austenitic steels. Identification of the Laves phases chemistry by microprobe analysis showed the precipitates in the matrix proper to be the Fe_2Ti type compound while the precipitates along grain boundaries are the Ti_4Fe_2O and/or Ti_2Ni type compounds; all precipitates were in the complex forms with possible substitutions among the Fe, Ni, and Co atoms, $(Fe, Ni, Co)_2$ Ti and Ti_2 (Fe, Ni, Co).

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LAVES PHASE FORMATION AND HARDENING OF
MARTENSITIC Fe-9Ni-9Co-Ti ALLOYS
Weily F. Chiao

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Technical Report AMMRC TR 83-19, May 1983, 27 pp-
illus-tables, D/A Project: 1T0611091A,
AMCMS Code: 611101-01-A84

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