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STRENGTH OF INITIALLY VIRGIN MARTENSITES AT -196 C AFTER AGING AND TEMPERING *

George T. Eldis and Morris Cohen

ABSTRACT

The compressive strength at -196 C of martensites in Fe-0.26%C-24%Ni, Fe-0.4%C-21%Ni, and Fe-0.4%C-18%Ni-3%Mo alloys, all with sub-zero Ms temperatures, has been determined in the virgin condition and after one hour at temperatures from -80 to +400 C. The effects of ausforming (20% reduction in area of the austenite by swaging at room temperature prior to the martensitic transformation) were also investigated. For the unausformed martensites, aging at temperatures up to 0 C results in relatively small increases in strength. Above 0 C, the age hardening increment increases rapidly, reaching a maximum at 100 C during the carbon clustering stage and prior to the precipitation of carbides. Above 100 C, the strength decreases continuously with increasing tempering temperature except for the molybdenum-containing alloy, which exhibits secondary hardening on tempering at 400 C. For the ausformed martensites, the response to aging at sub-zero temperatures is greater than for unausformed material. Strength again passes the a maximum on aging at 100 C. However, on tempering just above 10C C, ausformed materials show a slower rate of softening than the unausformed martensites. The strengthening produced by the ausforming treatment is largest for the Fe-0.4%C-18%Ni-3%Mo alloy, but there is no evidence of carbide precipitation in the deformed austenite to account for this effect of molybdenum.

 * Prepared for the Peter G. Winchell Symposium on Tempering of Steel, TMS-AIME Fall Meeting, 12-13 October, 1981, Louisville, KY.
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Massachusetts Institute of Technology, Cambridge, MA 02139.

INTRODUCTION

The strength of ferrous martensite has been the object of numerous investigations using many experimental techniques. One of the authors of the present study (M. Cohen) has reviewed the various strengthening mechanisms elucidated by prior work.¹ While that review is now several years old, a search of more recent literature indicates the review is still very much up-to-date, especially concerning the strength of martensite in the virgin state and in relatively lightly tempered conditions.

Much of the prior work has one limitation in common: Mechanical tests were performed at ambient temperatures. Thus, the strengthening, if any, due to the various ambient and sub-ambient temperature tempering reactions revealed by x-ray diffraction, 2,3 Mössbauer spectroscopy⁴ and resistometric techniques^{5,6} has generally not been investigated, and the strength of martensite in the virgin condition is still not well defined. The goal of the present study was to obtain a more accurate measure of the effects of various aging and tempering phenomena on the strength of ferrous martensites by eliminating, at the outset, any inadvertent aging prior to testing.

EXPERIMENTAL PROCEDURES

This study was undertaken concurrently with another investigation of martensite-tempering phenomena,⁶ and the general experimental procedures followed for preparation and heat treatment of the specimens are described in that work. The alloys selected for the present study were (nominally)

-2-

Fe-0.26%C-24%Ni, Fe-0.4%C-21%Ni, and Fe-0.4%C-18%Ni-3%Mo. Compositions and measured $M_{\rm S}$ temperatures of the alloys are shown in Table I.

Maintaining and testing martensitic specimens at liquid nitrogen temperatures was a paramount concern in this investigation. Therefore, compressive testing was used for all strength measurements. This procedure allowed the testing of a large number of specimens in a reasonable period of time compared with tensile testing, since there was relatively little difficulty in inserting refrigerated specimens into a refrigerated compressive test fixture and aligning the system for testing. The test specimens were of cylindrical geometry, 5 mm diameter by 12.5 mm long. They were loaded and strained several percent between hardened tool steel platens immersed in a liquid nitrogen bath. Load and crosshead displacement were measured and converted to engineering stress-strain data.

Prior work has shown that the martensitic substructure can play a significant role in various tempering phenomena.¹ Altering the substructure by deforming the austenite prior to refrigeration to form martensite was therefore also investigated here. This ausforming was accomplished by swaging at room temperature to nominally 20% reduction in area, the maximum deformation the alloys could withstand without the occurrence of some deformation-induced martensitic transformation. Both compressive and resistivity test specimens were subjected to these ausforming treatments. The procedure for measuring resistivity has been described elsewhere.⁶

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RESULTS AND DISCUSSION

Compressive flow stress values (0.6% offset) measured at -196 C after one hour of treatment at temperatures up to 400 C are shown in Figures 1-3 for the 26C-24Ni, 40C-21Ni and 40C-18Ni-3Mo alloys, respectively. Data are given for both "regular" martensite, that is, martensite produced from undeformed austenite, and for "ausformed" martensite which was generated from austenite that had been deformed approximately 20% at room temperature prior to the martensitic transformation. The reported flow stresses are for a mixture of 90 vol.-% martensite plus 10 vol.-% austenite which was obtained in the three alloys on refrigeration in liquid nitrogen. Experimental evidence presented in an appendix to this paper indicates that the 0.6% compressive flow stress is rather insensitive to retained austenite for contents less than approximately 15 vol.-%, and a correction for retained austenite is unnecessary in such instances.

The general features of the flow stress vs. aging/tempering curves are the same for all three alloys. For the initially virgin regular martensites, aging at temperatures up to 0 C results in relatively little change in the -196 C flow stress. Above 0 C, the flow stress rises rapidly; reaching a maximum after one hour at about 100 C. Tempering at higher temperatures results in a steady decrease in strength except for the molybdenum-containing alloy, which shows evidence of secondary hardening above 300 C. For the initially virgin ausformed martensite, there is again an increase in strength with increasing aging temperature, reaching a maximum at 100 C. However, the aging response of the ausformed martensite is somewhat more rapid at subzero temperatures than observed with the regular

-4-

martensites. On tempering above 100 C, the strength decreases, but the ausformed martensites remain stronger than the regular martensites. Hoffman⁷ and McEvily et al.,⁸ who employed much higher degrees of austenite deformation in their ausforming studies than did the present authors, have observed that ausforming increases the resistance to softening on tempering. The data of the present study also indicate improved temper resistance due to ausforming, as shown more clearly in Figure 4 and discussed further below.

Figures 1-3 also show the electrical resistivity of the alloys measured at -196 C after one hour of aging at various temperatures. The resistivity values have been corrected for the presence of retained austenite as described elsewhere.⁶ While these isochronal plots do not provide as much detailed information as isothermal plots and do not lend themselves well to the analysis of tempering kinetics, they nevertheless offer a convenient representation of the main changes observed on aging and tempering, and will suffice for the present discussion.

The resistivity data show all the responses to tempering previously noted in the literature:⁶ an initial decrease in resistivity, tentatively ascribed to the transformation of some retained austenite to martensite; an increase in resistivity to a maximum value, attributed to clustering of carbon atoms and the net increase in mean-square static displacement of the iron atoms accompanying this phenomenon; and a subsequent decrease in resistivity caused both by precipitation of carbides, which depletes the matrix of carbon, and by recovery of the martensitic substructure. Within this general framework of change in resistivity on tempering, several specific features are noteworthy. (1) As a result of ausforming, the magnitude of

-5-

the resistivity peak decreases. (2) Ausforming reduces the "rate" of decrease in resistivity from the peak value, especially in the 50 to 200 C range. (3) The addition of molybdenum to the material greatly reduces the magnitude of the resistivity peak. The first two observations are in accord with prior work⁷ where ausforming was accomplished by 75% reduction in area and the observed effects were therefore more pronounced. The effect of molybdenum in reducing resistivity peak height is in agreement with prior work on both chromium- and molybdenum-alloyed martensites.⁹

While not presented here, full isothermal records of resistivity vs. time at several temperatures were obtained for all three alloys and subjected to activation energy analysis as described elsewhere.⁶ The results were the same for all three alloys, whether ausformed or not. Through the resistivity peak (to about 100 C in terms of the isochronal plots in Figures 1-3), an activation energy of about 100 kJ/mole (24 kCal/mole), typical of carbon diffusion in martensite, was found. On further tempering, the activation energy increased, reaching a value of about 135 kJ/mole (33 kCal/mole), typical of iron-atom diffusion along dislocation paths during the precipitation of E-carbide and cementite (corresponding to the first and third stages of tempering).⁶

Figure 4 shows the difference in flow stress at -196 C between the ausformed and the regular martensites as a function of tempering temperature. Such a plot serves to accentuate differences in tempering response. The three alloys display two maxima, one near 0 C, reflecting the more rapid response to low-temperature aging of the ausformed martensite, and another

-6-

near 200 C, reflecting a greater resistance to softening of the ausformed martensites on tempering just beyond the peak strength in Figures 1-3. Between the two maxima, at a tempering temperature near 100 C which produces the maximum flow stress, the ausform-strengthening increment falls to the same level observed for virgin (unaged) martensite. The ausform strengthening increments for the two Fe-Ni-C alloys in Figure 4 are nearly equal for all tempering temperatures, and are about half the magnitude of the ausformstrengthening increments exhibited by the molybdenum-alloyed steel.

GENERAL DISCUSSION

It was previously shown⁶ that the increase in electrical resistivity with aging reflects a clustering of interstitial carbon atoms. For the regular martensites, the strengthening is most rapid in the same regime of time and temperature as the resistivity peak; but the maximum strength is reached at 100 C, well beyond the resistivity maximum, where the first stage of tempering (precipitation of ε -carbide) has started. Electron microscopy has revealed the presence of ε -carbide in these alloys after tempering one hour at 150 C,⁶ and the carbides were quite well developed at this stage.

The onset of carbide precipitation in the present work can be defined somewhat more precisely from observations in the literature; the data of Choo and Kaplow¹⁰ and of Hoffman⁷ are especially helpful in this regard. The alloy examined by Hoffman was nearly identical in composition to the 40C-21Ni alloy of this investigation, and so comparison with his work

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should be straightforward. Comparisons with the 1.86% C iron-carbon alloy of Choo and Kaplow must be regarded as more approximate because of the concentration-dependence of the activation energy for carbon diffusion in martensite.¹¹ Based on carbon diffusion as the rate-controlling step in the aging of martensite, up to the onset of carbide precipitation,⁶ one can calculate equivalent times at various temperatures to achieve the same observations as reported by Choo and Kaplow and by Hoffman, in order to compare more directly with the present findings. The results are given in Table II, and indicate that after 1 hour at 100 C (corresponding to the peak in strengthening) carbon-atom clusters are still present without any detection of ε -carbide (Choo and Kaplow), but after 1 hour at 114 C ε -carbide is beginning to form (Hoffman). It should also be noted that the strengthening peaks in Figure 1-3 are rather flat, signifying that the high strength levels which accompany the clustering process are maintained into the early stage of ε -carbide precipitation.

Ausforming 20% retards the softening of martensite on tempering at 200 C (Figure 4), where ε -carbide precipitation is unquestionably occurring in the regular martensite. Hoffman⁷ found that 75% ausforming has a much more pronounced effect than observed here; he also detected the enhanced temper resistance imparted by ausforming. In addition, after tempering ausformed specimens for three hours between 100 and 200 C, Hoffman's electron microscopy clearly revealed a marked suppression of ε -carbide precipitation in comparison with the regular martensite. Indeed, at the lower end of this tempering range there was no evidence of ε -carbide in the ausformed martensite, in marked contrast to the observations in regular martensite. These

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results on ausformed martensites lead us to conclude that the maximum strength attained on aging precedes the onset of carbide precipitation.

The experimental techniques (Mössbauer spectroscopy and transmission electron microscopy) which have furnished evidence of carbon atom clustering on aging have not yet been applied to ausformed martensites. However, the changes in the aging and tempering behavior resulting from ausforming are quite consistent with the proposed clustering process. They also imply some interesting differences between the transformation substructure (the substructure resulting from the martensite reaction itself) and the additional substructure derived from the ausforming process.

Following Basinski et al.¹² and Hoffman,⁷ we interpret the changes in resistivity on aging to be caused by changes in mean-square static displacements of the iron lattice atoms, including the substitutional alloy atoms. As shown in Figures 1-3, the initial resistivity is raised by ausforming, reflecting the increased mean-square displacements of the lattice atoms resulting from the strain fields of the inherited substructure. These strain fields will not, in general, be uniform. The atomic displacements will be largest at locations nearest the defects, e.g., near dislocation cores, and in these regions of high initial lattice distortion it is likely that interstitial atom clustering would result in relatively less <u>additional</u> lattice atom displacement than would occur elsewhere, in less strained regions of the lattice. Clustering in these highly strained regions would thus be expected to contribute less to the mean-square static displacements and, hence, to the resistivity peak, in accordance with the observed decrease in peak height as shown by the ausformed curves in Figures 1-3.

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As shown in Figure 4, the strengthening of the martensite due to ausforming is the same after aging to the maximum strength level at 100 C as it is for the martensite in the virgin condition. This additive character of the two strengthening mechanisms, hardening by the clusters and dislocation strengthening by the inherited substructure, implies the two mechanisms are independent, i.e., the clustering occurs to about the same degree regardless of the presence of the additional substructure from ausforming. However, the flow stress data in Figures 1-3 show that the inherited substructure does accelerate the aging response at sub-zero temperatures, indicating a propensity for the clusters to form more rapidly in the vicinity of the inherited dislocations, perhaps due to interaction of the strain fields surrounding both. Analysis of the resistivity data does not reveal any significant difference in the rate of clustering for ausformed martensite compared with regular martensite. This is not surprising, however. Resistivity is a measure of mean-square atomic displacements of the entire volume of material, while the volume of material significantly affected by the strain fields of the inherited dislocations would be a small fraction of the total volume, even for an inherited dislocation density as high as $10^{12}/cm^2$.

A typical substructure of ferrous martensite (the substructure resulting from the austenite-to-martensite transformation) is presumed to have a very high dislocation density, on the order of $10^{12}/\text{cm}^2$.¹ It is therefore noteworthy that the regular martensites do not display the same sub-zero aging response as their ausformed counterparts. This may indicate

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that the "regular" dislocations are arranged in some minimum energy configuration, interacting with one another in such a way that their net strain field is much less extensive than that due to the "inherited" dislocations.

The retardation of ε -carbide precipitation on tempering above 100 C which results from ausforming is also in accord with the carbon-clustering concept. The extra dislocations inherited from the ausforming apparently help stabilize the clusters with respect to the subsequent formation of ε -carbide. In earlier studies the binding energy of individual carbon atoms to dislocations was postulated to explain both the resolution of ε -carbide during strain tempering¹³ and the suppression of ε -precipitation after ausforming.⁷ By assuming enhanced clustering at "inherited" dislocations, the model of carbon affinity for dislocations is maintained, but more complex interactions among groups of atoms are involved.

The carbon-dependent strengthening of ferrous martensites has been treated by Winchell and Cohen¹⁴ as an interstitial solid-solution effect of well-separated carbon atoms in octahedral sites. The resulting increase in mean-square stress required to move a dislocation due to the iron-atom displacements was shown to account for the observed strengthening within a factor of two and for the observation that strength varies linearly with the one-third power of the carbon concentration. However, the carbon-dependent strength of martensite has been found to be quite insensitive to test temperature^{5,15} compared to what would be anticipated if strengthening were dependent on the interaction of dislocations with the stress fields surrounding individual carbon atoms. The present investigation now suggests that some

-11-

clustering may have already taken place prior to the strength measurements in previous work, and with the carbon-dependent strengthening then being due to groups of atoms, a reduced temperature sensitivity would be expected.

The strengthening response of the 40C-18Ni-3Mo alloy to aging suggests that the same basic phenomena are operative, at least up to the point where secondary hardening occurs in the molybdenum-containing alloy. The near absence of a resistivity peak in this material (Figure 3) can be explained in the same general way as the reduction in peak height due to ausforming. The substitution of 3 wt-% (about 1.75 at-%) Mo for 3 wt-% (about 3 at-%) Ni in a 0.4% C material increases the initial resistivity by about 19 microhm-cm (compare Figures 2 and 3). A similar resistivity increase would be expected for an increase in carbon content of about 0.7 wt-% (approximately 3.3 at-%).⁶ On the basis of the mean-square static displacement model for resistivity changes, it is clear that the molybdenum atoms severely distort the lattice. The additional lattice displacements caused by clustering in an already distorted lattice should be small, thus resulting in little accompanying resistivity increase.

As shown in Figure 4, the "efficiency" of the ausforming operation, i.e., the martensite strengthening achieved by a given amount of austenite deformation at room temperature, is largest in the molybdenum-alloyed material. This enhanced response to ausforming is in accord with previous observations on steels containing alloy carbide-formers.¹⁶ It has been ascribed to the precipitation of alloy carbides during austenite deformation, leading in turn to increased strain hardening and dislocation generation in the austenite and, hence, to a higher dislocation density in the martensite subsequently

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formed.^{17,18} No evidence was found for such carbide precipitation in the deformed austenite of the 40C-18Ni-3Mo alloy investigated here, neither in the stress-strain behavior of the fully austenitic material nor in examination of thin foils of the austenite after deformation.¹⁹ Thus, in the present experiments at least, molybdenum exerts its influence on ausform-strengthening by some mechanism other than carbide precipitation.

SUMMARY

1. Aging of virgin martensites for one hour at temperatures in the range of 0 to 100 C results in significant strengthening attributable to the clustering of carbon atoms. Small amounts of strengthening are detectable at temperatures as low as -80 C. Maximum strength is attained after aging at 100 C, prior to detectable carbide precipitation.

2. Ausformed virgin martensites undergo more rapid strengthening on aging at sub-zero temperatures compared to the regular virgin martensites, but the total age-hardening increment achievable on aging to maximum strength is not much affected by ausforming. Here again, the maximum strengthening precedes detectable carbide precipitation. The additional dislocations derived from ausforming appear to enhance the rate of cluster formation and stabilize the clusters with respect to carbide precipitation.

3. Both ausforming and alloying with molybdenum in the Fe-Ni-C martensites investigated here diminish the peak in electrical resistivity which accompanies the clustering of carbon on aging. In both cases, the

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indications are that ausforming and molybdenum contribute significantly to the mean-square lattice-atom displacements and hence to the electrical resistivity of virgin martensites. Thus during subsequent aging, the effect of carbon atoms on the lattice atom displacements and resistivity changes is relatively less pronounced.

4. The greater ausform-strengthening achievable in steels alloyed with carbide-forming elements noted in the literature is confirmed with the molybdenum-containing alloy examined here, but deformation-induced precipitation of carbides in the austenite, previously thought responsible for the improved response to ausforming, is precluded under the experimental conditions of the present work.

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REFERENCES

- 1. M. Cohen: Trans. JIM, 1968, Vol. 9 (supplement), pp. XXIII-XXIX.
- L. I. Lysak and Ya. N. Vovk: <u>Phys. Met. Metallog.</u>, 1965, Vol. 19, No. 5, pp. 52-58.
- 3. P. C. Chen and P. G. Winchell: Met. Trans., 1980, Vol. 11A, pp. 1333-1339.
- N. De Cristofaro, R. Kaplow and W. S. Owen: <u>Met. Trans.</u>, 1978, Vol. 9A, pp. 821-825.
- 5. P. G. Winchell and M. Cohen: Trans. ASM, 1962, Vol. 55, pp. 347-361.
- A. M. Sherman, G. T. Eldis and M. Cohen: "The Aging of Iron-Nickel-Carbon Martensite," this symposium.
- D. W. Hoffman: "Ausform-Strengthening of Iron-Nickel-Carbon Martensite," Ph.D. Thesis, Massachusetts Institute of Technology, 1966.
- A. J. Mc Evily, Jr., R. H. Buck, F. W. Schaller and D. J. Schmatz: Trans. ASM, 1963, Vol. 56, pp. 753-767.
- 9. H. W. King and S. G. Glover: J.I.S.I., 1960, Vol. 196, pp. 281-288.
- 10. W. K. Choo and R. Kaplow: Acta Met., 1973, Vol. 21, pp. 725-732.
- 11. M. Hillert: Acta Met., 1959, Vol. 7, pp. 653-658.
- Z. S. Basinski, J. S. Dugdale and A. Howie: <u>Phil. Mag.</u>, 1963, Vol. 8, pp. 1989-1997.
- 13. D. Kalish and M. Cohen: Mater. Sci. Eng., 1970, Vol. 6, pp. 156-166.

References (Continued)

- 14. P. G. Winchell and Morris Cohen: <u>Electron Microscopy and Strength of</u> <u>Crystals</u>, G. Thomas and J. Washburn, eds., pp. 995-1007, Interscience, New York, 1963.
- 15. M. J. Roberts and W. S. Owen: J.I.S.I., 1968, Vol. 206, pp. 375-384.
- G. Thomas, D. Schmatz and W. Gerberich: <u>High Strength Materials</u>, V. F. Zackay, ed., pp. 251-297, Wiley, New York, 1965.
- R. Phillips and W. E. Duckworth: <u>High Strength Materials</u>, V. F.
 Zacka, ed., pp. 307-326, Wiley, New York, 1965.
- 18. O. Johari and G. Thomas: <u>Trans. ASM</u>, 1965, Vol. 58, pp. 563-578.
- G. T. Eldis: "Ausforming and Tempering of High Alloy Steels," Ph.D. Thesis, Massachusetts Institute of Technology, 1971.

References (Continued)

- 14. P. G. Winchell and Morris Cohen: <u>Electron Microscopy and Strength of</u> <u>Crystals</u>, G. Thomas and J. Washburn, eds., pp. 995-1007, Interscience, New York, 1963.
- 15. M. J. Roberts and W. S. Owen: J.I.S.I., 1968, Vol. 206, pp. 375-384.
- G. Thomas, D. Schmatz and W. Gerberich: <u>High Strength Materials</u>, V. F.
 Zackay, ed., pp. 251-297, Wiley, New York, 1965.
- R. Phillips and W. E. Duckworth: <u>High Strength Materials</u>, V. F.
 Zackay, ed., pp. 307-326, Wiley, New York, 1965.
- 18. O. Johari and G. Thomas: Trans. ASM, 1965, Vol. 58, pp. 563-578.
- G. T. Eldis: "Ausforming and Tempering of High Alloy Steels," Ph.D. Thesis, Massachusetts Institute of Technology, 1971.

Table I

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COMPOSITIONS (WT-%) AND M_S TEMPERATURES (°C) OF ALLOYS STUDIED

				Mn.						
Alloy	ပ	ï	£	P, N	s	SI	บ	A	e F	Å
26C-24Ni	0.26	23.94	<0.01	<.001	0.005	0.049	0.002	0.002	bal.	-2
40C-21Ni	0.40	21.14	<0.01	<.001	0.005	0.049	0.002	0.001	bal.	-7
0C-18Ni-3Mo	0.38	17.50	2.85	<.001	0.001	0.051	0.002	0.011	bal.	-5

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Table II

TIME - TEMPERATURE REGIMES FOR CLUSTERING AND &-CARBIDE FORMATION

Source	Observation	Time - Temperature	Approximate Equivalent Time - Temperature
Choo and Kaplow (Reference 10)	Clusters present	2160 h at 25 C	5 h - 82 C 1 h - 100 C
Choo and Kaplow (Reference 10)	Clusters gone, ɛ present	2160 h at 25 C plus 62 h at 82 C	67 h - 82 C 15 h - 100 C 1 h - 137 C
Hoffman (Reference 7)	e forming	3 h at 100 C	1 h - 114 C
Hoffman (Reference 7)	ɛ well developed	3 h at 150 C	ł

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FIGURE CAPTIONS

- Resistivity (Dashed Curves) and Compressive Flow Stress (0.6% Offset, Solid Curves) Measured at -196 C after Tempering One Hour at Indicated Temperatures. Alloy 26C-24Ni.
- Resistivity (Dashed Curves) and Compressive Flow Stress (0.6% Offset, Solid Curves) Measured at -196 C after Tempering One Hour at Indicated Temperatures. Alloy 40C-21Ni.
- Resistivity (Dashed Curves) and Compressive Flow Stress (0.6% Offset, Solid Curves) Measured at -196 C after Tempering One Hour at Indicated Temperatures. Alloy 40C-18Ni-3Mo.
- Difference in Flow Stress Between Ausformed and Regular Martensites, Measured at -196 C after Tempering One Hour at Indicated Temperatures.
- A-1. Strength of Fe-Ni-C Martensite-Austenite Mixtures as a Function of Volume-Percent Martensite. Hoffman: Tensile Tests, 0.8% Offset, Samples Tempered and Tested at 30 C. Winchell: Compressive Tests, 0.6% Offset, Samples Tempered and Tested at 0 C. This Work: Compressive Tests, 0.6% Offset, Samples Tempered as Indicated and Tested at 23 C.

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Figure 2 Resistivity (Dashed Curves) and Compressive Flow Stress (0.6% Offset, Solid Curves) Measured at -196 C after Tempering One Nour at Indicated Temperatures. Alloy 40C-21Ni.

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Figure 3 Resistivity (Dashed Curves) and Compressive Flow Stress (0.6% Offset, Solid Curves) Measured at -196 C after Tempering One Hour at Indicated Temperatures. Alloy 40C-18Ni-3Mo.



Figure 4 Difference in Flow Stress Between Ausformed and Regular Martensites, Measured at -196 C after Tempering One Hour at Indicated Temperatures.

APPENDIX

Effect of Retained Austenite Content on the Strength of Martensite-Austenite Mixtures

In the present investigation, quenching the alloys in liquid nitrogen resulted in a retained austenite content of 10 ± 1 vol-%. The austenite content was determined by measuring the saturation magnetization of the test specimens after aging one hour at room temperature. The sensitivity of this magnetic method was estimated to be better than ± 1 vol-% austenite. Full details of the test procedure are given elsewhere.¹⁹

Previous work by Winchell et al.⁵ and Hoffman⁷ revealed a linear variation of strength with martensite for martensite contents \leq 85 vol-%, in accord with a simple rule of mixtures:

$$\sigma = \sigma_{\rm M} V_{\rm M} + \sigma_{\rm Y} (1 - V_{\rm M}) \tag{A-1}$$

Here, σ and V are flow stress and volume fraction, respectively, and the subscripts γ and M refer to the austenite and martensite constituents.

In the present work, it was possible to achieve martensite contents as high as 95 vol-% by refrigerating the specimens in liquid helium. A brief investigation was therefore undertaken to determine if a linear relationship between strength and martensite content would be maintained to such high martensite levels.

Compressive specimens were prepared and refrigerated to various temperatures to produce 80 to 95 vol-% martensite. The specimens were warmed to room temperature and aged one hour. Retained austenite content was measured and the specimens were either immediately subjected to compressive testing or given an additional one-hour temper at 100 to 400 C prior to testing. Compressive tests were performed at room temperature, to better facilitate comparison with prior work^{5,7} which employed test temperatures of 0 or 30 C.

Results are presented in Figure A-1 for the 40C-21Ni alloy as curves of flow stress vs. martensite content. Data of Winchell et al., who also employed compressive testing, and Hoffman, who employed tensile tests, are shown for comparison. Both investigators also studied Fe-Ni-C alloys. The present work shows a marked leveling off of the strength vs. martensite content curves at martensite contents in excess of 85%.

The reason for the decrease in the sensitivity of strength to austenite content as the amount of martensite increases can be understood simply from Equation A-1. Differentiating, we obtain

$$\frac{d\sigma}{dV_{\rm M}} = \sigma_{\rm M} - \sigma_{\rm Y} \tag{A-2}$$

The data in Figure A-1 indicate the quantity $\sigma_M \cdot \sigma_\gamma$ decreases as V_M increases. While σ_M would not be expected to be a function of V_M , σ_γ should be expected to increase, due to the work hardening that occurs as a result of the martensitic transformation itself. This work hardening is evident from transmission electron microscopy which clearly shows that untransformed austenite becomes highly dislocated as adjacent regions transform to martensite. Table A-1, which presents the results of microhardness readings (5 g load) on some specimens from this study, also demonstrates the work hardening of the austenite.

Table A-1

Hardness (HV5) of the Various Microconstituents in the 40C-21Ni Alloy After Aging at Room Temperature

Microconstituent	Hardness	Approximate Equivalent Tensile Strength, [*] MPa (ksi)
Austenite in fully austenitic material	214	·
Martensite, in 80% martensite - 20% austenite mixture	600	2000 (290)
Austenite, in 80% martensite - 20% austenite mixture	540	1800 (260)

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*by conversion from ASTM tables

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ပံ Strength of Fe-Ni-C Martensite-Austenite Mixtures as a Function of Volume-Percent Martensite. Compressive Tests, 0.6% Offset, Samples Tempered as Indicated and Tested at 23 Compressive Tests, 0.6% Offset, Samples Tempered and Tested at 0 C. Tensile Tests, 0.8% Offset, Samples Tempered and Tested at 30 C. This Work: Winchell: Hoffmen: Figure A-1

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to room temperature and aged one hour. Retained austenite content was measured and the specimens were either immediately subjected to compressive testing or given an additional one-hour temper at 100 to 400 C prior to testing. Compressive tests were performed at room temperature, to better facilitate comparison with prior work^{5,7} which employed test temperatures of 0 or 30 C.

Results are presented in Figure A-1 for the 40C-21Ni alloy as curves of flow stress vs. martensite content. Data of Winchell et al., who also employed compressive testing, and Hoffman, who employed tensile tests, are shown for comparison. Both investigators also studied Fe-Ni-C alloys. The present work shows a marked leveling off of the strength vs. martensite content curves at martensite contents in encess of 85%.

The reason for the decrease in the sensitivity of strength to austenite content as the amount of martensite increases can be understood simply from Equation A-1. Differentiating, we obtain

$$\frac{d\sigma}{dV_{\rm M}} = \sigma_{\rm M} - \sigma_{\rm Y} \qquad (A-2)$$

The data in Figure A-1 indicate the quantity $\sigma_N - \sigma_\gamma$ decreases as V_N increases. While σ_M would not be expected to be a function of V_M , σ_γ should be expected to increase, due to the work hardening that occurs as a result of the martensitic transformation itself. This work hardening is evident from transmission electron microscopy which clearly shows that untransformed austenite becomes highly dislocated as adjacent regions transform to martensite. Table A-1, which presents the results of microhardness readings (5 g load) on some specimens from this study, also demonstrates the work hardening of the austenite.

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