Effect of Aging Treatment on the Mechanical Properties of C-250 Maraging Steel by Flow Forming

I.K. Lee, C.P. Chou, C.M. Cheng, and I.C. Kuo

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The technique of forward flow forming has been used to produce a long, thin walled tube made of C-250 maraging steel. The forward flow forming can save raw material, increase strength, and reduce the production process time. Because the work hardening effect on solution-treated C-250 using flow forming is minimal, the flow-formed tube requires an additional heat treatment to obtain higher hardness and strength. With the direct aging treatment, low elongation values are obtained, making this treatment unsuitable for the engineering design. It was found that the 540 °C/6 h/AC over-aging treatment results in better strength and elongation values. The strengthening phase of the flow-formed C-250 maraging steel was found to be the intermetallic compound of Fe_3Mo.

Keywords aging treatment, flow-forming, maraging steel C-250

1. History

The 18% nickel maraging steel owes its outstanding characteristics to certain specific features of the iron-nickel based alloys. The martensitic structure with a low rate of distortion can be attributed to the extra-low carbon (< 0.03 wt.%) content. In the solution-annealed state, after air cooling from the elevated temperatures, a body-centered cubic (bcc; α-phase) structure is present[1,2,3] and it can be easily shaped using conventional processes such as rolling, forging, and flow forming.[3] To strengthen the maraging steel, a high temperature aging treatment is required. The main hardening precipitates of intermetallic compounds include Ni_3Mo, Ni_3Ti, Fe_2Mo, or FeTiMo.[2,3,4]

The technique of forward flow forming has been used to produce a long, thin-walled tube of C-250 maraging steel. Flow forming can produce parts nearer to net shape than forging, eliminate some initial rough machining, conserve material, increase strength, and reduce the production process time.[5,6] Due to the minimal work hardening effect of solution-treated C-250 by the flow-forming process,[1] the flow-formed tube requires an additional aging treatment to obtain higher hardness and strength.[4]

2. Materials

The material used was C-250 maraging steel which has been double vacuum melted using vacuum induction melting-vacuum air remelting (VIM-VAR) according to AMS 6512C.[7] The chemical composition of C-250 maraging steel was measured by scanning electron microscopy (SEM)/energy dispersive x-ray analysis (EDX) and carbon analysis. The results are presented in Table 1. After solution treating at 815 °C for 1 h, the preforms were cold forward flow-formed from 8.0-1.7 mm by one pass. The reduction rate of thickness was 78.8%.

Table 1 Chemical Composition of C-250 Maraging Steel, wt.%

Element	Specifications AMS 6512 C	Used in This Work	A(a)	B(a)
Al	0.05 ~ 0.15	0.15	0.44	0.00
Mo	4.6 ~ 5.2	4.9	12.95	4.63
Ti	0.3 ~ 0.5	0.5	0.38	0.44
Co	7.0 ~ 8.5	8.6	6.05	8.36
Ni	17 ~ 19	17.6	16.74	16.95
C	0.03	0.003	…	…
Cr	<0.50	…	…	…
Cu	<0.5	…	…	…
Fe	Bal.	Bal.	63.43	69.62

(a) Chemical compositions of Fig. 10b

3. Experimental Methods

To increase the strength after flow forming, several types of additional heat treatments were performed. The flow-formed tubes were heat treated in three aging conditions: under-aged (370 °C/10 h/AC, 480 °C/1 h/AC); aged (480 °C/3 h/AC, 480 °C/6 h/AC); and over-aged (540 °C/6 h/AC, 580 °C/6 h/AC), as shown in Table 2. The flow chart of the experiments schedule is shown in Fig. 1.

The microstructures of flow-formed and aged samples of the preform were observed. The positions for microstructural analysis were in the cross section of the flow-formed tube wall. Specimens were polished to 0.005 μm, etched with 30 ml HCl + 10 ml HNO_3 + $CuCl_2$, and examined using an optical microscope. Hardness was measured on the Vickers hardness scale with 300 g/15 s. The scale was then auto-transformed to a Rockwell C scale (HRC). The microstructures of the flow-

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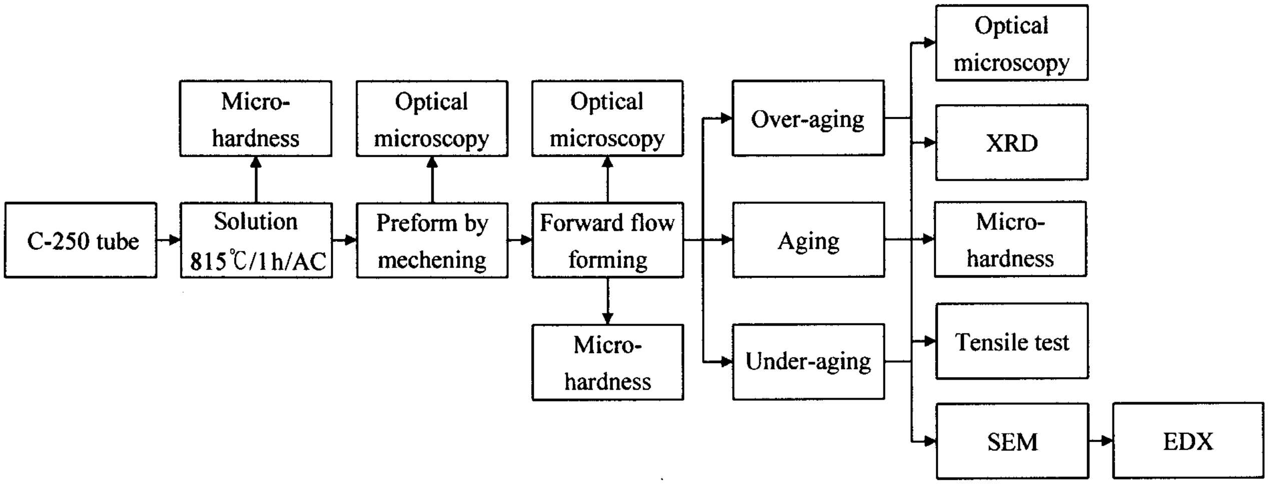


Fig. 1 The flow chart of experiments of C-250 maraging steel

Table 2 Mechanical Properties of Aging Treated Flow Formed C-250 Maraging Steel in Different Aging Treatments

Sample No.	Heat Treating Process	YS, MPa, (0.2% Offset)	UTS, MPa	Elongation, %
370-1	Solution → forming	1018.0	1145.0	5.1
370-2	Solution → forming → aging 370/10h	1529.0	1565.0	1.9
370-3	Solution → forming → aging 480/1h	1763.0	1795.0	1.1
370-4	Solution → forming → aging 480/3h	1956.0	1977.0	1.6
370-5	Solution → forming → aging 480/6h	1930.0	1954.0	1.6
370-6	Solution → forming → aging 540/6h	1800.0	1884.0	3.6
370-7	Solution → forming → aging 580/6h	1528.0	1620.0	5.2

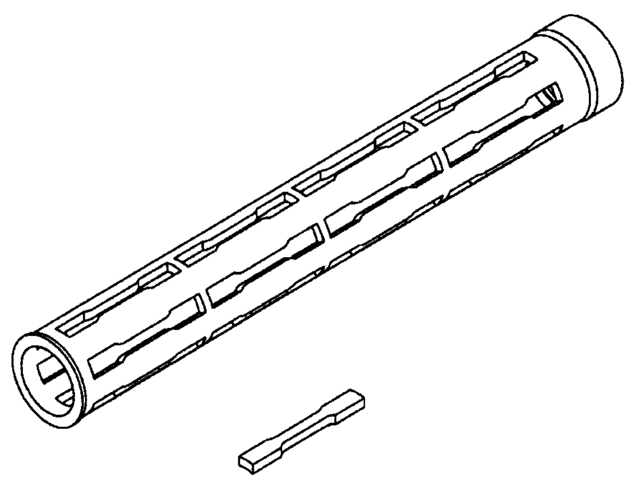
formed samples were identified by x-ray diffraction (XRD) using a Siemens Diffractometer D5000 (Germany), with a Cu-K_α beam source (λ = 1.5418 Å). The scanning angle range covered from 40-90° and the scanning speed was 1°/min.

The tensile specimens were made following guidelines from ASTM standard E370 by milling along the axis direction of the tube (Fig. 2). The gage length was 50.8 mm. The strain rate was 0.2 mm/min before yield strength (YS) and 2 mm/min after YS. Six specimens were tested for each condition. Fractographic observations of tensile-tested samples in as-formed and aged condition were made in a 20 kV SEM to determine the failure modes. EDX of fragmented particles was used to analyze the chemical composition.

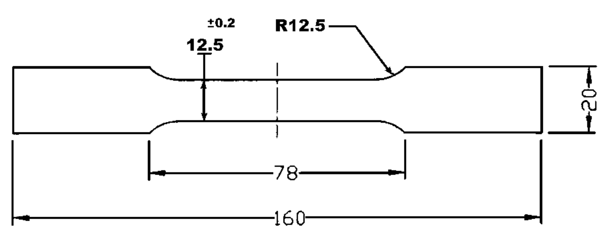
4. Discussion and Results

The result of chemical composition analysis is shown in Table 2. All elements are within the range of standard C-250 (AMS 6512C) except cobalt (Co), which is higher than the standard value.[7]

Figure 3 shows the optical micrographs of C-250 maraging steel before and after flow forming. The C-250 preform treated by solution-annealing treatment is shown in Fig. 3(a). The fine equiaxed crystal was clearly seen in the matrix phase. No standard lath martensite structure was seen in the microstructure.[3] Figure 3(b) indicates that α-phase was precipitated in the matrix and no deformed grains were observed.



(a)



(b)

Fig. 2 Dimensions of tensile specimen

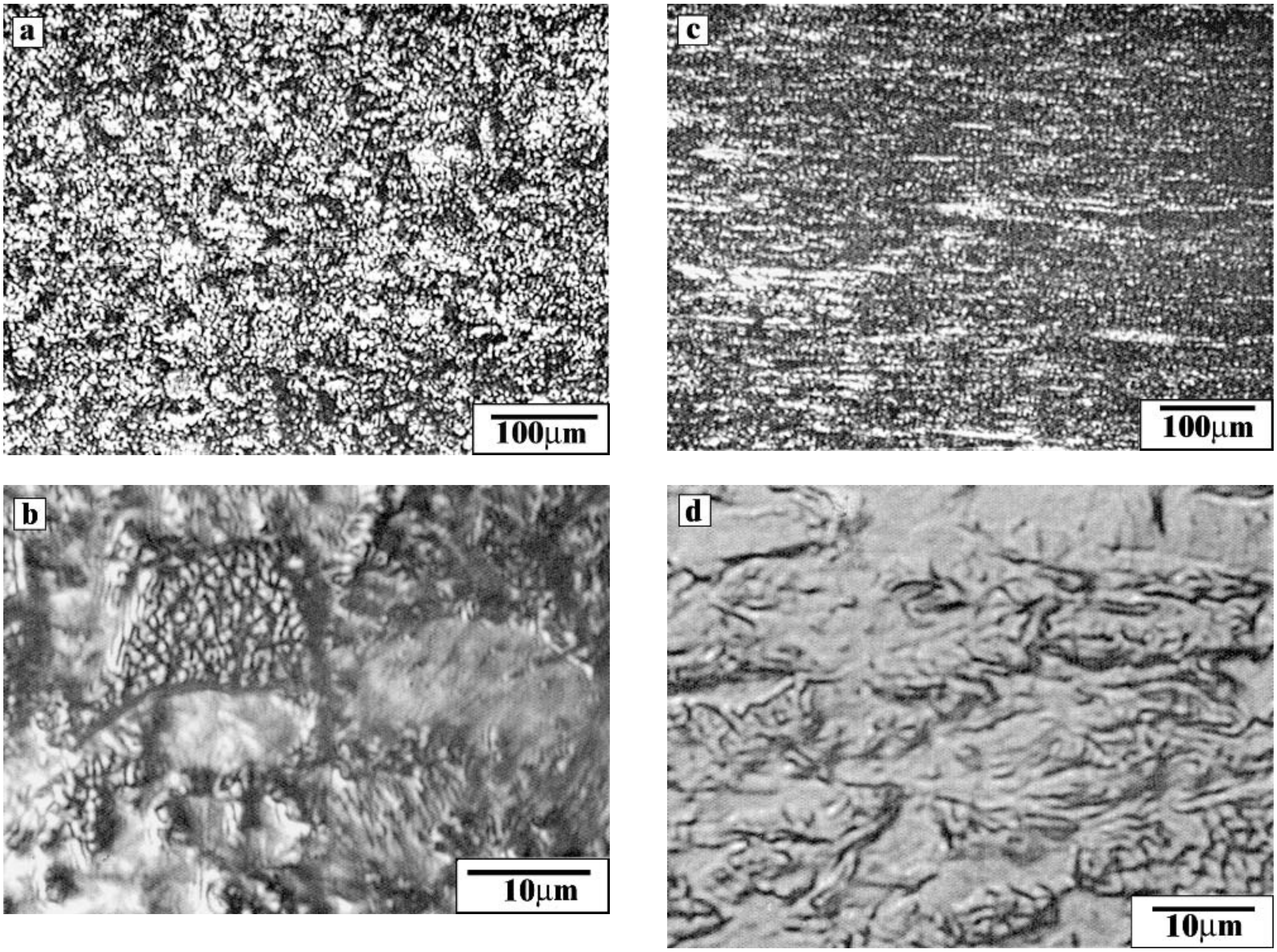


Fig. 3 Optical micrograph of C-250 maraging steel: **(a)** and **(b)** preform; **(c)** and **(d)** after flow forming

The microstructure of flow-formed (8.0 t → 1.7 t) C-250 maraging steel is shown in Fig. 3(c). The average microhardness of the outside part is 35.1 HRC after work-hardening, an increase of 4.9 HRC scale. The microhardness of the inner part of the formed tube is 32 HRC. This difference in hardness indicates that the effect of cold working for forward flow forming is not uniform, as shown in Fig. 4. The flow-formed tube was driven by forming stress in both the axial and radial directions. Under the stress of forming, the microstructure was distorted and an extended grain shape is observed (Fig. 3d).

The microstructures of the flow-formed tube after two different aging treatments are shown in Fig. 5. The hardness was increased to 51.6 HRC after a single aging treatment and 52.1 HRC after a double aging treatment. These values were about the same as the standard values of the testing material. After the aging treatment, the hardness of flow-formed C-250 maraging steel was increased to almost twice that of the as-formed steel; however, the microstructure had no obvious change as shown in Fig. 3(c) and 5.

The results of tensile tests of the flow-formed tube of C-250 maraging steel in the as-formed, under-aged, aged, and over-aged conditions are presented in Table 2.

According to AMS specification 6520C [8] of C-250 maraging steel, the standard values are as follows: yield strength (YS)—1689 MPa; ultimate tensile strength (UTS)—1758 MPa; elongation rate (Gage 50.8 mm)—2.5%. The YS and UTS of specimen 370-4 are 15.8% and 12.5% higher than the standard values, respectively, but the elongation is lower than that of the standard value. With the standard aging treatment condition on the formed tube, the combined effect of work-hardening and precipitation-hardening should enhance the tensile strength.

According to Floreen's suggestion,[3] the over-aging treatment can be used to reduce the strength and increase the elongation rate in two ways: (1) with the standard treatment temperature of 480 °C, the aging time should be extended to over 100 h; and (2) with a higher aging treatment temperature.

After flow forming, the mechanical properties of C-250 are dependent on the aging time (Fig. 6). In the 480 °C/1 h/AC condition, the tensile strength of the formed tube cannot reach the maximum value from aging precipitation, but the elongation drops to the lowest as indicated in Fig. 6. The newly precipitated intermetallic compounds may prevent the future dislocation slip; therefore, the tensile strength is increased and

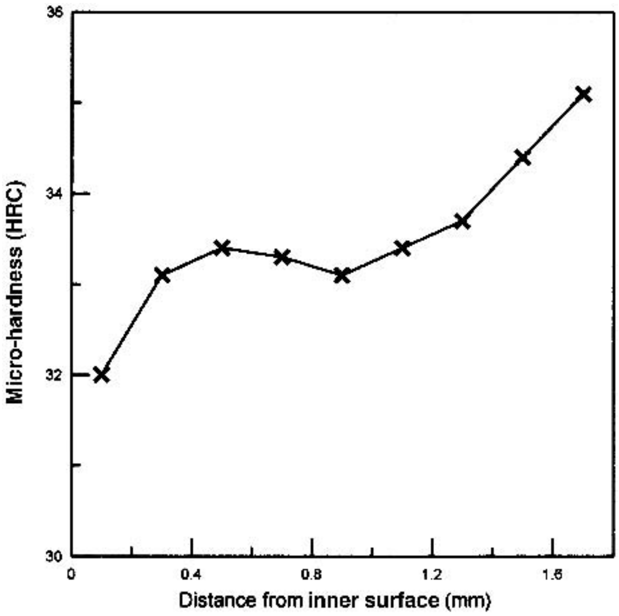


Fig. 4 The hardness distribution in the cross section of wall by flow-formed tube of C-250 maraging steel

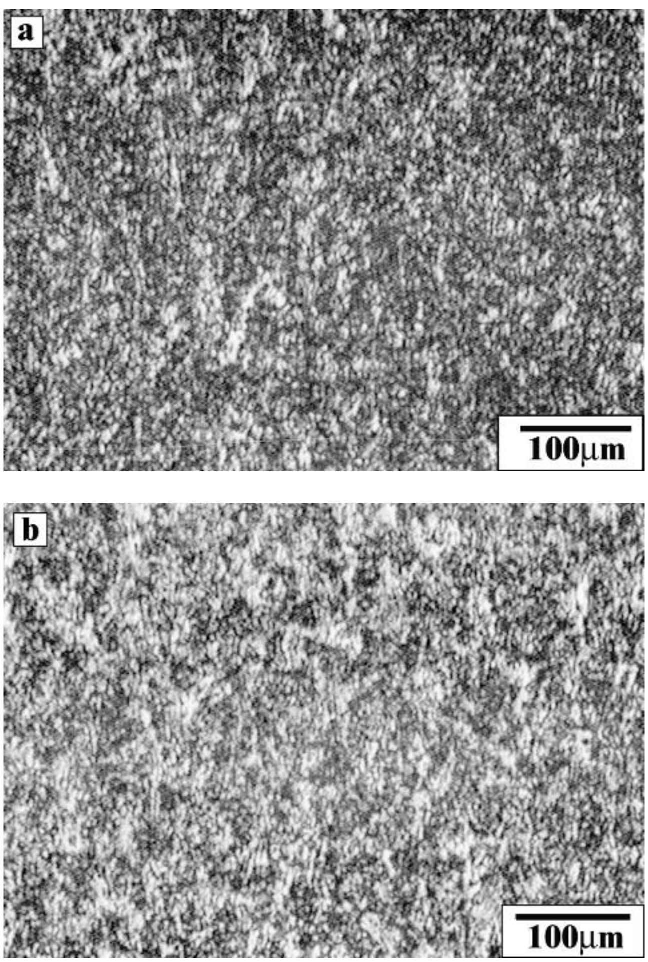


Fig. 5 Optical micrographs of flow-formed C-250 maraging steel after (**a**) 480 °C/6 h/AC; and (**b**) 480 °C/6 h/AC +480 °C/6 h/AC

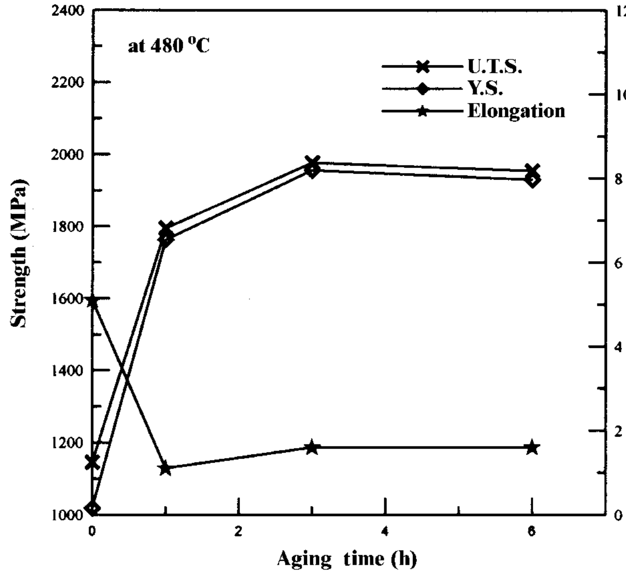


Fig. 6 Mechanical properties versus aging time of C-250 flow-formed maraging steel

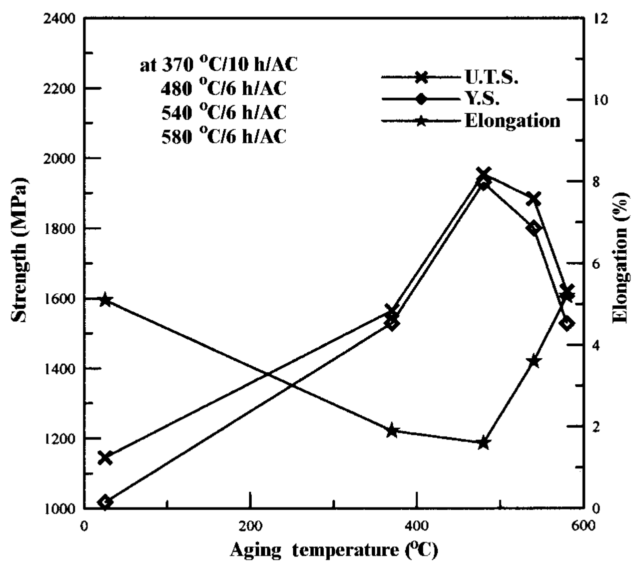


Fig. 7 Mechanical properties versus aging temperature of C-250 flow-formed maraging steel

the elongation rate is decreased. As the standard aging time was extended to 6 h (370-5), the value of tensile strength was similar to that of 370-4. This result indicates that the aging temperature 480 °C can produce stable results. This is consistent with the results of other reports.[3]

Figure 7 shows the effect of aging temperature on the mechanical properties of flow-formed C-250 maraging steel. It was shown that the tensile strength increased smoothly when the aging temperature was under 370 °C. The maximum value of the tensile strength can be achieved as the temperature was increased to 480 °C. However, the elongation rate was reduced to the lowest value. When the aging temperature was increased, the strength dropped. At the higher temperature for 6 h, the

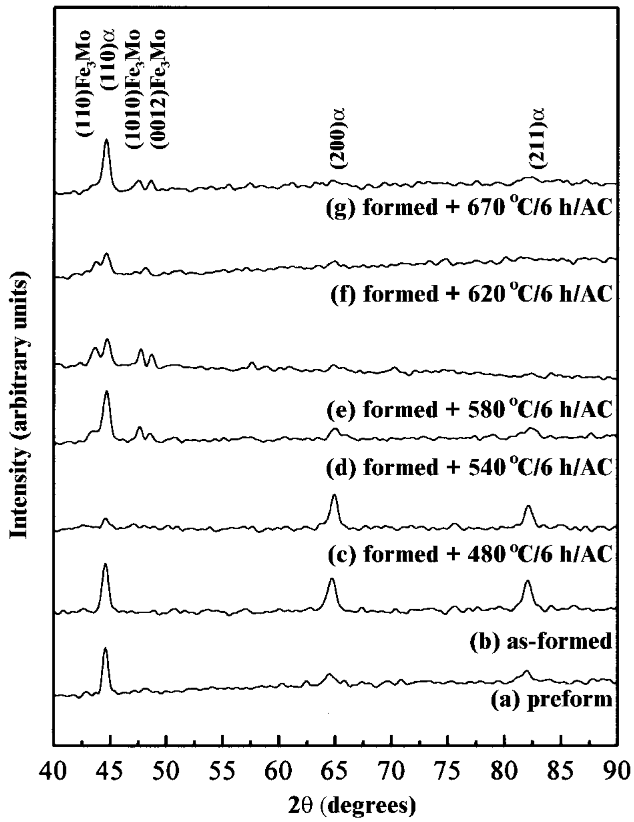


Fig. 8 X-ray diffractogram of C-250 maraging steel. **(a)** preform, **(b)** as-formed, and flow formed +, **(c)** 480 °C/6 h, **(d)** 540 °C/6 h, **(e)** 580 °C/6 h, **(f)** 620 °C/6 h, **(g)** 670 °C/6 h

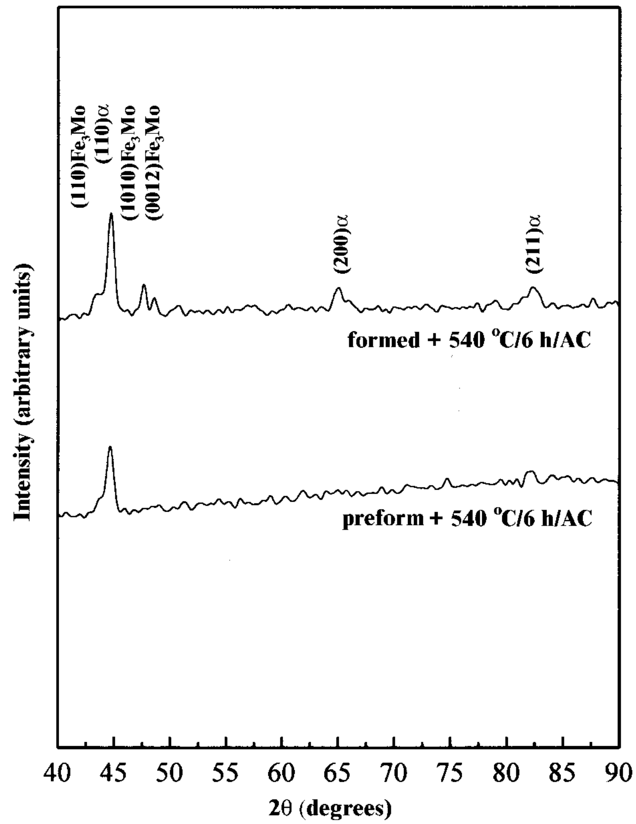


Fig. 9 X-ray diffractogram of C-250 maraging steel: preform + 540 °C/6 h and formed 540 °C/6 h

strength obtained reached the value of the over-aging treatment. The high temperature (HT) enhanced the diffusion rate, the aged precipitates were clustered, and grain coarsening occurred. Also, at HT the solid solubility of alloy elements was higher in the matrix-phase and the amount of aged precipitates was decreased. As the aging temperature was increased to over 500 °C, the strength was further decreased and the elongation increased. This heat treatment can cause the martensite to transform into softer austenite and is called "austenite reversion."[3,9] With much higher temperature aging treatments, the behavior of the reversion became more complete. Figure 7 shows that the elongation rate increased from 1.6% at 480 °C to 3.6% at 540 °C and up to 5.2% at 580 °C.

The XRD spectra obtained from the preform, as-formed, and as-aged (from 480-670 °C) samples are shown in Fig. 8. It has been observed that the preform of C-250 maraging steel usually transforms into bcc martensite at room temperature after air-cooling from 815 °C (Fig. 8a). The relative peak intensities of the preform sample corresponded to the values expected of a homogeneous and equiaxed material (Fig. 3b). The maximum intensity in the preform condition is observed at the $(110)_\alpha$ peak position. In the as-formed state, the maximum intensity is also at the $(110)_\alpha$ peak position. But the increase in the $(200)_\alpha$ and $(211)_\alpha$ peak intensities was also observed in the as-formed state. The changes observed in the relative peak intensities are undoubtedly due to the incorporated texture.[4] These results are similar to the results obtained by Ali et al.[10] They found that the relative intensities of the x-ray spectra start to change when reduction ratios were above 50 for flow-formed 18Ni 350 grade maraging steel.

To study the effects of aging on the texture, as-formed samples were aged at various temperatures. The XRD spectra obtained from formed plus aging specimens are also shown in Fig. 8. The (110), (1010), and (0012) peaks of Fe_3Mo phase were first detected in samples aged at 540 °C for 6 h (Fig. 8d). The relative (110) Fe_3Mo peak intensity of the aged samples increases as the aging temperature increases from 540-620 °C. However, the strengthening phases of maraging steel are formed by heterogenous precipitation, and these phases have been identified as intermetallic compounds of the Ni_3(Ti, Mo) and Fe_2Mo types.[11] A general formula of the type (Ni, Fe, $Co)_3$(Ti, Mo) has been assigned to these precipitates. The concentration of Mo and Ti in the precipitates are dictated by the relative amounts of these elements present in the alloy.[11] It has been shown[11,12] that the Ni and Ti atom in the Ni_3Ti unit cell can be replaced by the Fe and Mo atom, respectively. The A_3B precipitates with a hexagonal close-packed (cph) structure are favored due to the better lattice fit with the bcc martensite.[11] Flow forming can enhance the precipitation of intermetallic Fe_3Mo phase as shown in Fig. 9.

Figure 10 shows typical SEM fractographs of tensile-tested sample in as-formed condition. Examination of the fracture surface indicates the presence of nonuniform dimples (Fig. 10a). The larger dimples could be associated with the initiation

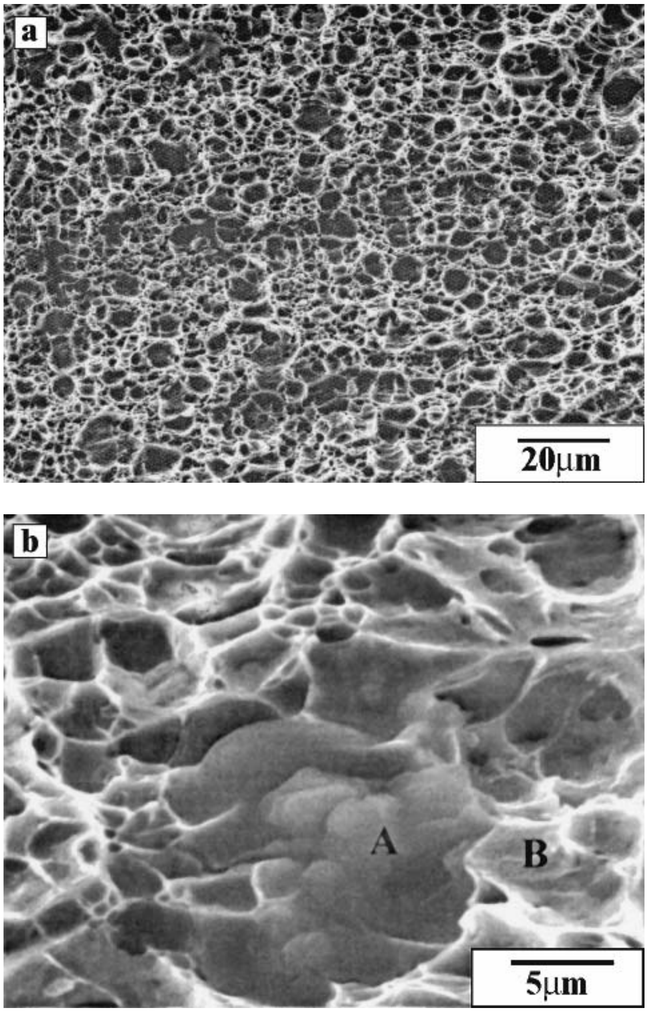


Fig. 10 Scanning electron fractographs of tensile tested in as-formed condition: **(a)** non-uniform dimples; **(b)** inclusions in the larger dimples

of crack on the primary inclusion (Fig. 10b). The chemical compositions of the inclusion are Al-rich and Mo-rich as analyzed by EDX (Table 2). The small dimples could only be attributed to the participation of precipitates as sites for crack generation.[13]

Aging at 480 °C for 6 h (370-5) resulted in a mixture of fine dimple and quasicleavage rupture in the present work (Fig. 11). In general, the features in this case are relatively coarser and exhibit larger deformation. This could be related to possible formation of austenite phase boundaries, to blunt a propagating crack.[14] However, Floreen and Decker reported[15] that plastic deformation can increase the amount of reverted austenite formed before the aging treatment. Ali et al. studied[10] the 18Ni 350 grade maraging steel for aging temperature up to 680 °C and found that the reverted austenite content for a particular aging temperature generally increases with the degree of deformation. The austenite phase appears along the martensite boundaries, but it is difficult to get a representative analysis of these thin layers of austenite by using XRD method.[16] Because a softer phase is present in a harder matrix,[13] the softer

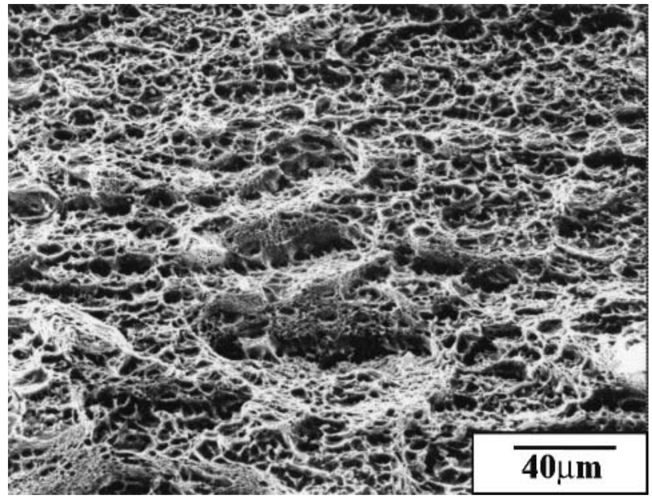


Fig. 11 Scanning electron fractograph of tensile-tested sample in 480 °C/6 h

phase will reach the critical strain of fracture at an earlier stage during loading. This would lead to the generation of a series of microcracks in a harder martensitic matrix.

5. Conclusions

The following are the main conclusions:

1) After 78.8% reduction by a flow-formed and a standard aging treatment, the C-250 maraging steel tube has a higher strength and lower elongation than the values of AMS specification 6520 C. This is due to the combined effect of work hardening and age hardening. Those values are not suitable to the design requirements.
2) To get appropriate mechanical properties of the flow-formed C-250 maraging steel, longer aging times (>6 h) can be used at an aging temperature of 480 °C.
3) To obtain a better elongation rate for the flow-formed C-250 maraging steel tube, the aging temperature can be increased to 540 °C for the over-aged condition. In this treatment, the austenite reversion transformation can take place. With an aging treatment of 540 °C/6 h/AC, a suitable strength value and elongation rate can be obtained. The strengthening phase of the flow-formed C-250 maraging steel was the intermetallic compound of Fe_3Mo.

Acknowledgments

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