Influence of Banded Structure on the Mechanical Properties of a High-Strength Maraging Steel

M. Ahmed, I. Salam, F.H. Hashmi, and A.Q. Khan

Chemical inhomogeneity results in the formation of banded structure in high-strength maraging steels. Segregation of titanium and molybdenum was found to be the primary cause of banded structure formation. When the concentrations of these elements increased beyond certain critical levels, bands comprising different grain sizes formed. The inclusions existed preferentially along the interface of the bands. A high-temperature homogenization treatment substantially reduced or eliminated the banded structure. The large grain size resulting from the homogenization treatment was subsequently reduced by a grain refinement treatment. The mechanical properties of the steel substantially improved following homogenization and grain refinement.

Keywords

banded structure, grain refinement, heat treatment, homogenization, mechanical properties

1. Introduction

THE BANDED structure occurs quite commonly in high-strength maraging steels. Its presence has been attributed to the segregation of alloying elements during the solidification process. In subsequent forming operations such as extrusion or cold rolling, regions exhibiting segregation extend along the material flow direction. The different response to etching solutions results in the appearance of bands.

Several workers have investigated the banded structure present in maraging steels. Goldberg (Ref 1) has suggested that carbon in combination with phosphorus can lead to the formation of banded structure, and Salmon Cox et al. (Ref 2) have shown that segregation of titanium, molybdenum, and nickel can also produce banding. This article examines the role of chemical inhomogeneity in forming the banded structure in maraging steel grade 18Ni(350) (2400 MPa), the effectiveness of heat treatment in reducing or eliminating the banded structure, and its influence on mechanical properties.

2. Experimental Method

The stock material was received in the form of 40 mm diam extruded rods with the chemical composition given in Table 1. The banded structure could be revealed by preparing samples using conventional metallography techniques and etching with 3% nital solution. To enhance the grain size distribution, the samples were electrolytically etched using 10% chromic acid solution. Homogenization and grain refinement were carried out in an air furnace; the heat treatment cycle is shown in Fig. 1. Homogenization temperature and time varied from 1200 to 1300 °C and 2 to 8 h, respectively. Charpy impact and tensile strengths were measured using standard samples prepared as per ASTM specifications; because of the size of the stock material, measurements were limited to the extrusion direction. Scanning electron microscopy (SEM) was used to study the fracture surfaces.

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3. Results and Discussion

Two types of banded structures were observed along the extrusion direction. The first was the conventional type frequently encountered in high-alloy steels. The segregation of alloying elements produces dark and white contrast as a consequence of different etching behaviors (Fig. 2). The bands run along the direction of material flow, their width and pattern varying with the degree of inhomogeneity and the mechanical processing history.

The white and dark regions were analyzed at several different locations and in different samples. The average compositions are given in Table 2, which shows that the white regions

Table 1 Composition of the stock material

Element	Weight percent
Nickel	17.9 ± 0.3
Cobalt	11.9 ± 0.3
Molybdenum	4.1 ± 0.3
Titanium	1.3 ± 0.1
Aluminum	<0.15
Carbon	≤0.003
Sulfur	≤0.005
Iron	bal

Table 2 Composition of white and dark bands
Average of 30 analysis points obtained from different regions of the bands

	Weight percent	
Element	White band	Dark band
Nickel	18.1 ± 0.3	17.3 ± 0.5
Cobalt	11.9 ± 0.1	12.1 ± 0.2
Molybdenum	4.5 ± 0.3	4.2 ± 0.3
Titanium	1.5 ± 0.1	1.2 ± 0.1
Aluminum	<0.1	<0.1
Iron	bal	bal

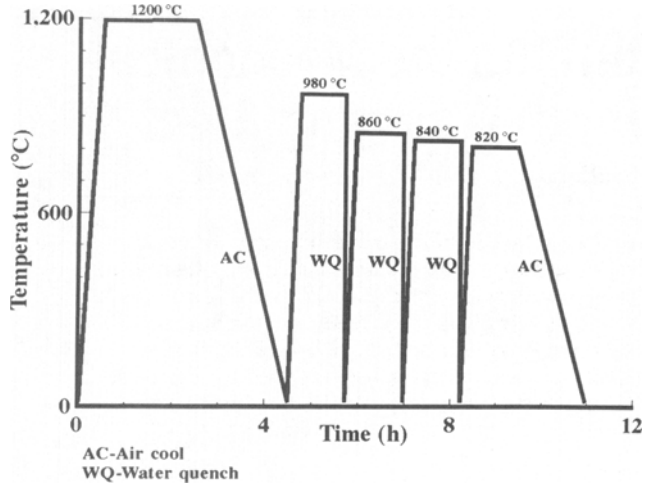


Fig. 1 Heat treatment cycle for homogenization and grain refinement

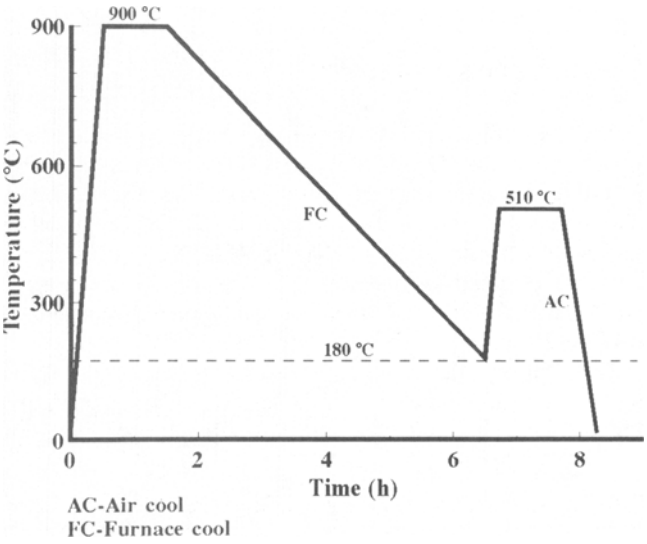


Fig. 3 Heat treatment cycle for selective aging

were enriched in nickel, molybdenum, and titanium. The carbon concentration could not be analyzed because of detector window thickness limitations, and the sulfur level was below the detectable limit of energy-dispersive x-ray analysis in SEM. The carbon and sulfur levels in the material are quite low (Table 1). The banded structure is probably not related to these elements.

The presence of banded structure and its relationship to segregation can be clearly elucidated by subjecting the samples to the selective aging treatment developed by Goldberg (Ref 1). The heat treatment cycle used in the present study is depicted in Fig. 3. This treatment promotes selective transformation of austenite to martensite along the bands where M_s is relatively higher. Subsequent aging at 510 °C induces precipitation along the transformed regions, whereas the austenite remains unaffected. When the sample is cooled after aging, the microstructure comprises aged and unaged martensite. Upon etching, the banded structure is clearly delineated, as can be seen in the optical micrographs presented in Fig. 4(a) and (b). Figure 4(c) shows a SEM image obtained from the aged martensite. The morphology appears to be different from the lath martensite observed in unaged martensite. Goldberg (Ref 1) also observed this, labeling it a "weblike" morphology. The variation in chemical composition is the most likely reason for the formation of weblike martensite (Ref 3).

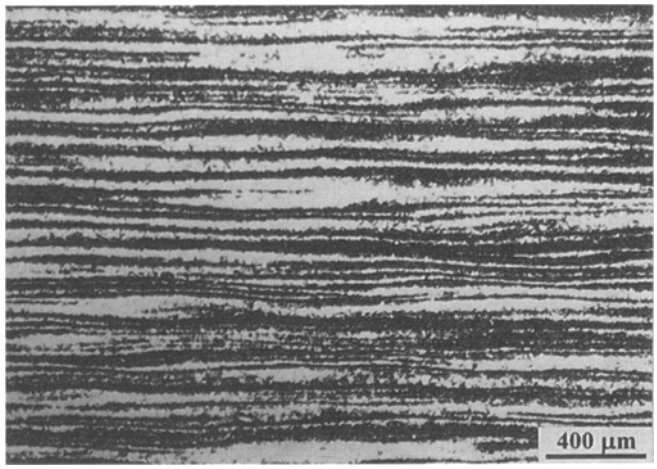


Fig. 2 Dark and white contrast produced after etching

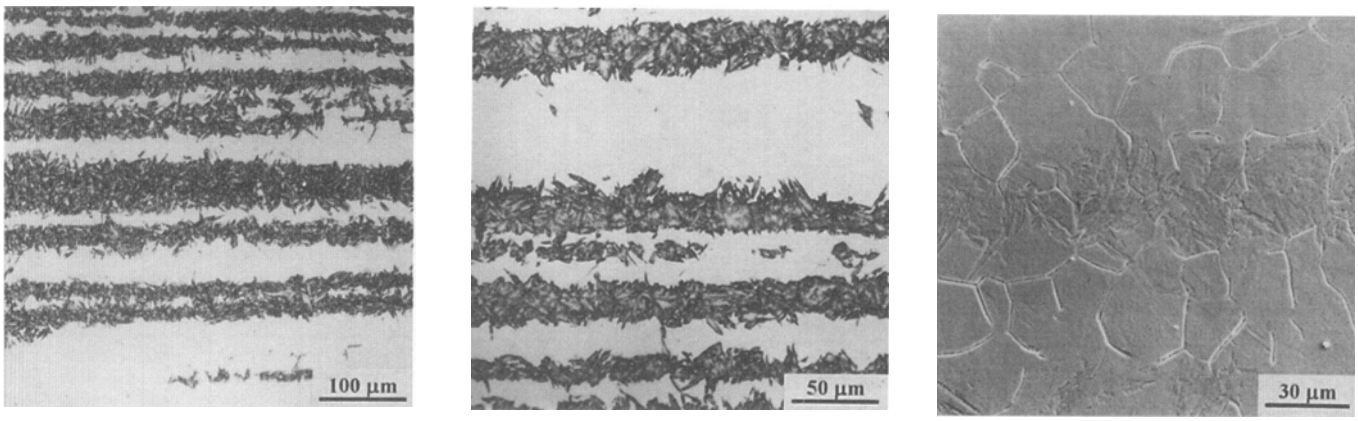
Table 3 Composition of coarse and fine grains
Average of 30 readings from different locations and several different rods

Element	Weight percent	
	Coarse grains	Fine grains
Nickel	18.3 ± 0.3	17.4 ± 0.4
Cobalt	11.9 ± 0.3	11.9 ± 0.2
Molybdenum	4.6 ± 0.2	3.9 ± 0.3
Titanium	1.6 ± 0.1	1.2 ± 0.1
Aluminum	0.15 ± 0.05	0.1 ± 0.1
Iron	bal	bal

3.1 *Grain Size Variation*

A second type of banded structure, consisting of different grain sizes, was observed along the extrusion direction. Figure 5 is an optical micrograph showing the microstructures along the three planar sections. The difference in size between large and small grains varied from sample to sample. On average, the coarse grains were three and a half to four times larger than the fine ones. The average compositions of the large and small grains are given in Table 3.

On the basis of these results, it can be concluded that the variation in grain size also resulted from the nonhomogeneity present in the material. Segregation appears to have influenced the recrystallization temperature and the subsequent grain growth following hot extrusion. The hardness of coarse and fine grains was measured as 385 ± 10 and 340 ± 10 HV, respectively. The partitioning of titanium and molybdenum was sufficient to cause the hardness difference observed. Nickel and molybdenum are very strong austenite stabilizers (Ref 4). When segregation increases beyond a certain level, the M_s tem-



(a) (b) (c)

Fig. 4 (a) and (b) Bands revealed after selective aging of the samples. (c) SEM image showing the weblike morphology of the aged martensite

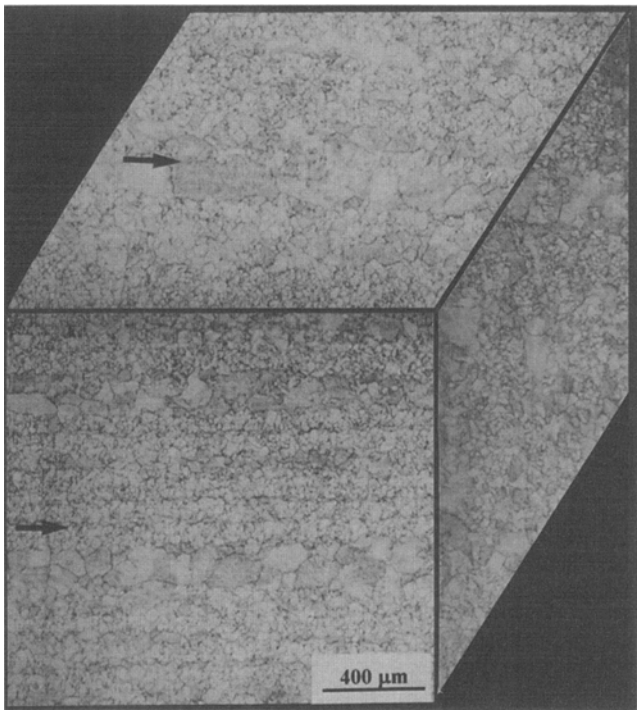


Fig. 5 Optical micrograph showing the microstructures along the three planar sections

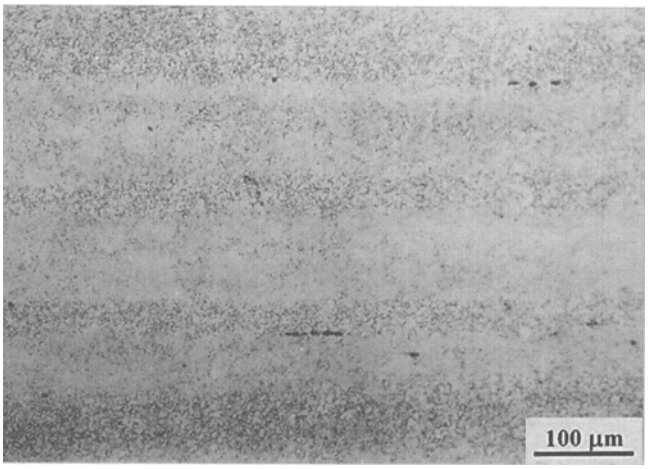


Fig. 6 Optical micrograph showing inclusions along the interface of coarse and fine grains

perature rises above the ambient value. The banded structure consisting of martensite and austenite observed by Salmon Cox et al. (Ref 2) may have been caused by an even higher level of segregation of nickel and molybdenum.

Thus, we can conclude that the degree of segregation determines the type of banded structure produced. It can change from the commonly observed dark and white bands to bands of variable grain size. At even higher levels of segregation, bands consisting of martensite and austenite may form.

In several samples, inclusions were located preferentially along the interface of coarse and fine bands (Fig. 6). The role of inclusions, if present, in terms of the second type of banded structure is not yet understood. However, a string of inclusions can introduce anisotropy in mechanical properties when measured along and across the extrusion direction. This could not be ascertained due to size limitations of the test materials.

3.2 *Homogenization Treatment*

Specimens were subjected to a homogenization treatment to reduce the concentration gradient of alloying elements present in different bands. The temperatures and times were fixed between 1200 and 1300 °C and for 2 to 8 h, respectively. The microstructure following homogenization is shown in Fig. 7, which reveals that the banded structure is substantially reduced or eliminated. However, homogenization substantially increased the grain size. The average grain size after homogenization treatment for different time intervals and temperatures is shown in Fig. 8.

It was also observed that scale losses (i.e., scale that chips off or can be removed by scrubbing) at temperatures above 1200 °C were approximately 5 to 7% by weight. Preferential oxidation of the prior austenite grain boundaries resulted in additional material degradation (Fig. 9a). The thickness of scale that remained adherent to the sample after scrubbing is plotted

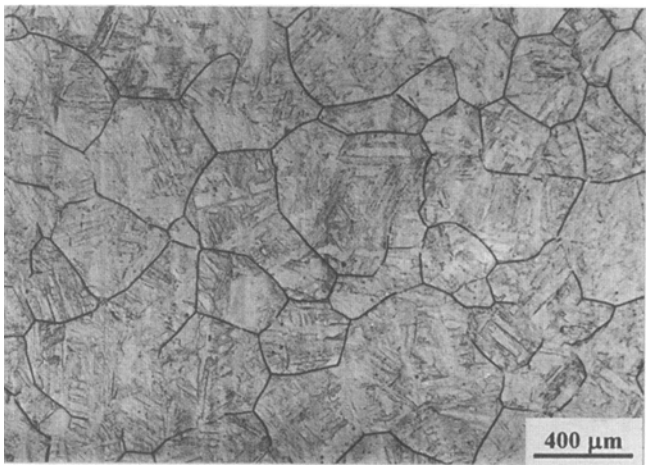


Fig. 7 Microstructure of the specimen after homogenization treatment

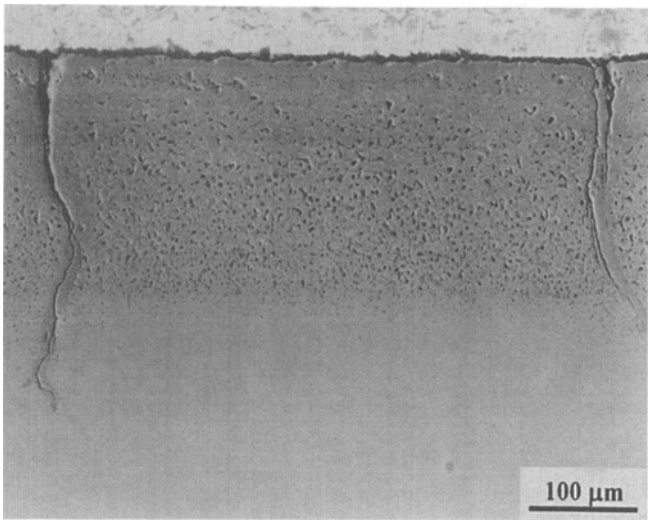
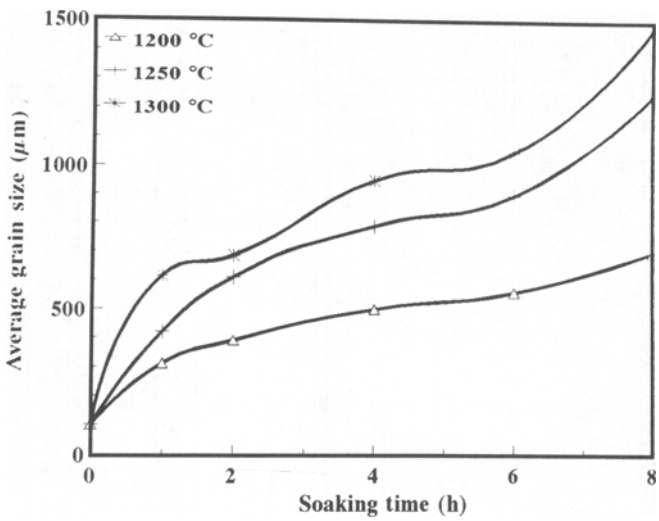
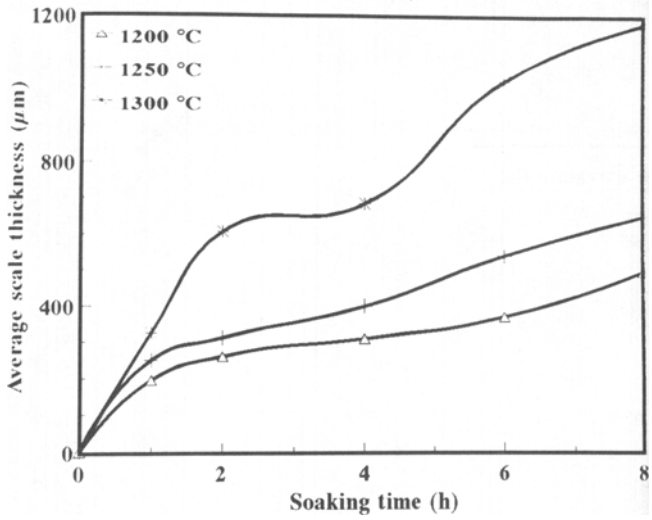


Fig. 8 Average grain size after homogenization treatment



(a)



(b)

Fig. 9 (a) Oxidation of the prior austenite grain boundaries. (b) Average scale thickness after homogenization treatment

in Fig. 9b. To keep the scale losses within practical limits, most of the work thus was done at 1200 °C.

3.3 *Grain Refinement*

As mentioned earlier, the homogenization treatment resulted in the formation of very large grains. This phenomenon can be lessened by subjecting the material to a grain refinement (GR) treatment. The heat treatment cycle incorporating homogenization and grain refinement is illustrated in Fig. 1. The grain sizes obtained following different treatments are listed in Table 4.

3.4 *Sample Size Effect*

The GR data in Table 4 were obtained from samples measuring 8 by 8 by 4 mm that were homogenized at 1200 °C. When the same treatment was applied to sections suitable for making Charpy and tensile specimens (i.e., 15 by 15 by 120 mm), the refinement was limited to the outer few millimeters. The inner section comprised mainly coarse grains (Fig. 10). The area fraction of the coarse grains lessened as the homogenization time was increased. The nonhomogeneity in grain size, therefore, is related to the soaking time during homogenization. A critical minimum homogenization time (depending on workpiece thickness) is necessary to achieve a fine distribution of grains following the GR treatment. Figure 11 depicts the effect of homogenization time on grain size distribution, showing average grain size as a function of distance from the center.

The area fraction of unrefined or large grains was also measured and is depicted as a function of homogenization time in Table 5. These data demonstrate that the homogenization time is an important variable that must be established for each case, depending on the material and the degree of segregation present in the bands. In the present case, homogenization treat-

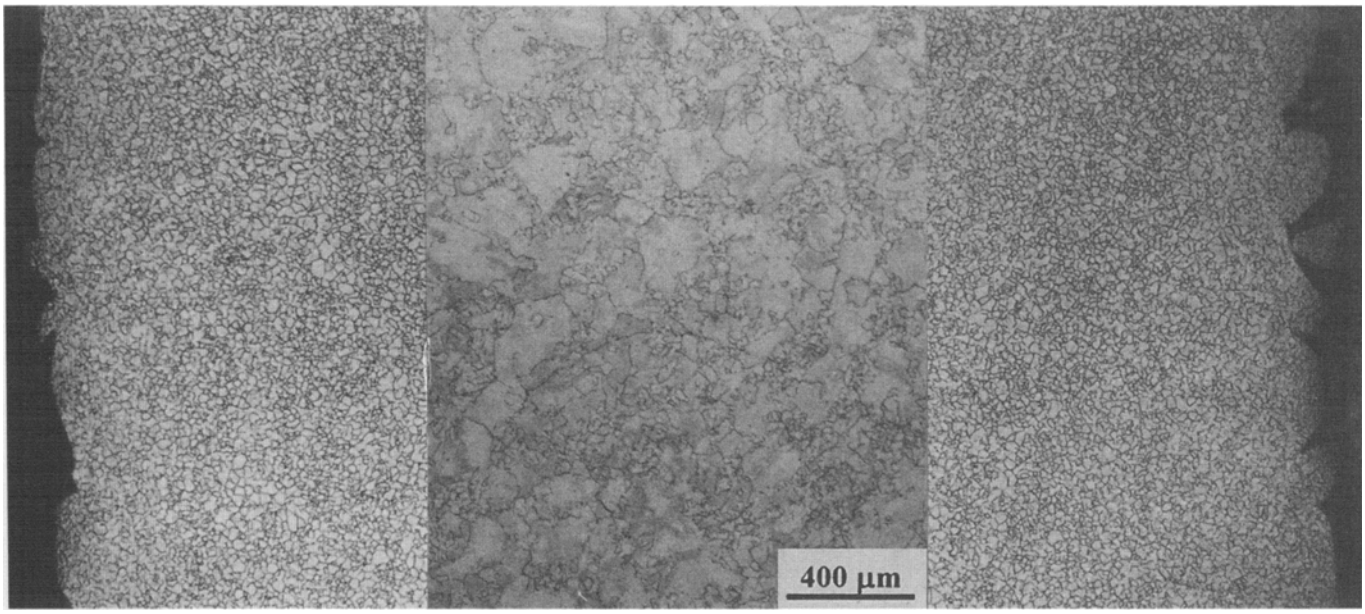


Fig. 10 Variation in grain size along the thickness of the specimen

Table 4 Grain sizes resulting from various heat treatments

Heat treatment(a)	Grain size, μm
1200 °C/2 h AC	309 ± 132
1200 °C/2 h AC, 980 °C/1 h WQ	67 ± 28
1200 °C/2 h AC, 980 °C/1 h WQ, 860 °C/1 h WQ	25 ± 9
1200 °C/2 h AC, 980 °C/1 h WQ, 860 °C/1 h WQ, 840 °C/1 h WQ	16 ± 6
1200 °C/2 h AC, 980 °C/1 h WQ, 860 °C/1 h WQ, 840 °C/1 h WQ, 820 °C/1 h AC	16 ± 6

(a) The sample size was fixed at 8 by 8 by 4 mm. AC, air cool; WQ, water quench

Table 5 Effect of homogenization time on area fraction of coarse grains

Heat treatment	Area fraction of coarse grains
1200 °C/2 h + GR	0.68
1200 °C/4 h + GR	0.38
1200 °C/6 h + GR	0.15
1200 °C/8 h + GR	0.10

ment at 1200 °C for 8 h produces uniform grain size across the specimen.

The influence of heat treatment on tensile strength, elongation, and reduction in area was also investigated. The data presented in Table 6 clearly show that mechanical properties improve when the segregation effect, depicted in the form of banded structure, is eliminated. The improvement in tensile strength may not be substantial, but reasonable increases in percent elongation and reduction in area are obtained, indicating that overall quality and flow properties of the material have improved. The grain sizes of the as-received and heat-treated material were of the same order; therefore, the improvement is directly related to the reduction of microsegregation present in the stock material.

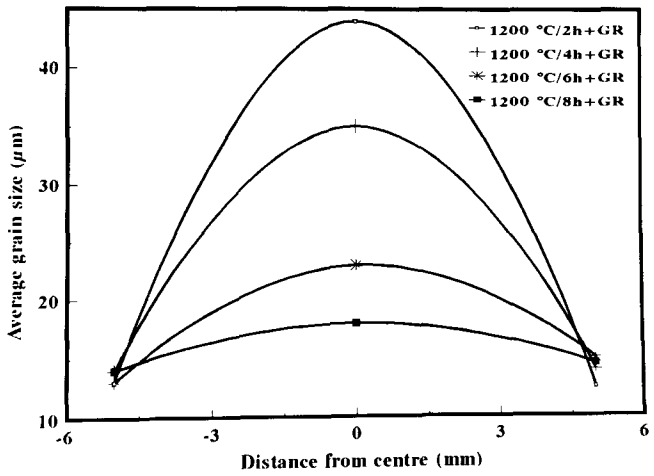


Fig. 11 Effect of homogenization time on grain size distribution

The change in material characteristics could also be seen when fractured surfaces were examined using SEM. The fractographs obtained from the original stock material are presented in Fig. 12. The low-magnification image (Fig. 12a) shows the primary fracture area surrounded by the shear lip ring. The image obtained from the primary fracture area (Fig. 12b) shows dimples of different sizes and shapes. Some of the dimples are quite large and elongated, surrounded by a network of fine dimples. It appears that strings of inclusions initiated the crack, resulting in elliptically shaped dimples (Fig. 12c). As mentioned earlier, the inclusions were found preferentially between coarse and fine grain bands. These most likely contributed to the formation of the elongated dimples. The presence of banded structure is also visible in the shear lip area (Fig. 12d).

The fractographs obtained following homogenization and grain refinement are presented in Fig. 13. The low-magnification image (Fig. 13a) shows the high degree of reduction in area

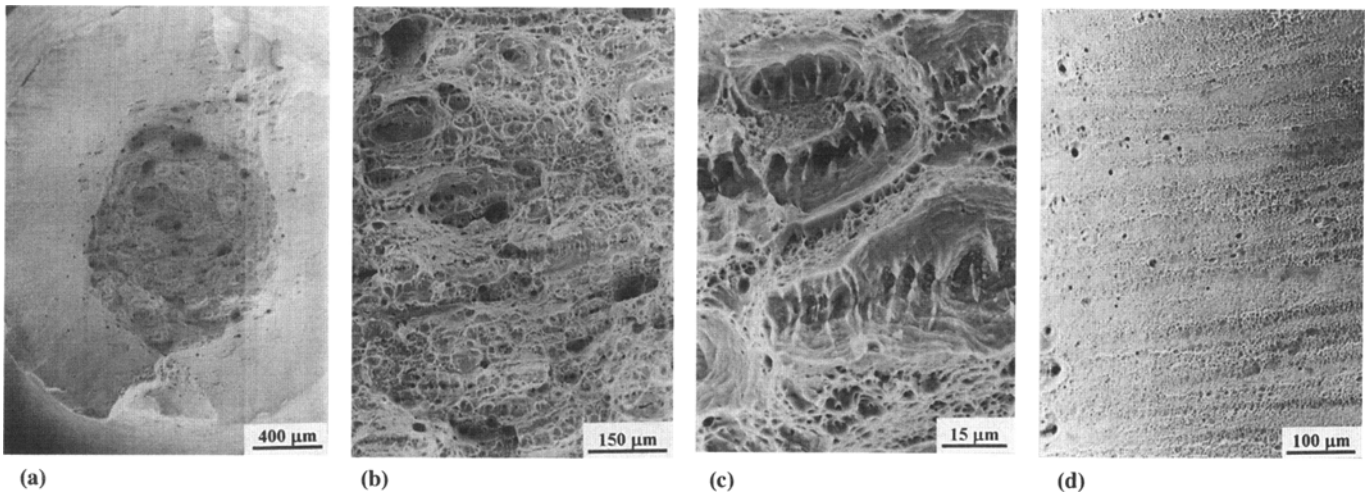


Fig. 12 SEM fractographs of the stock material

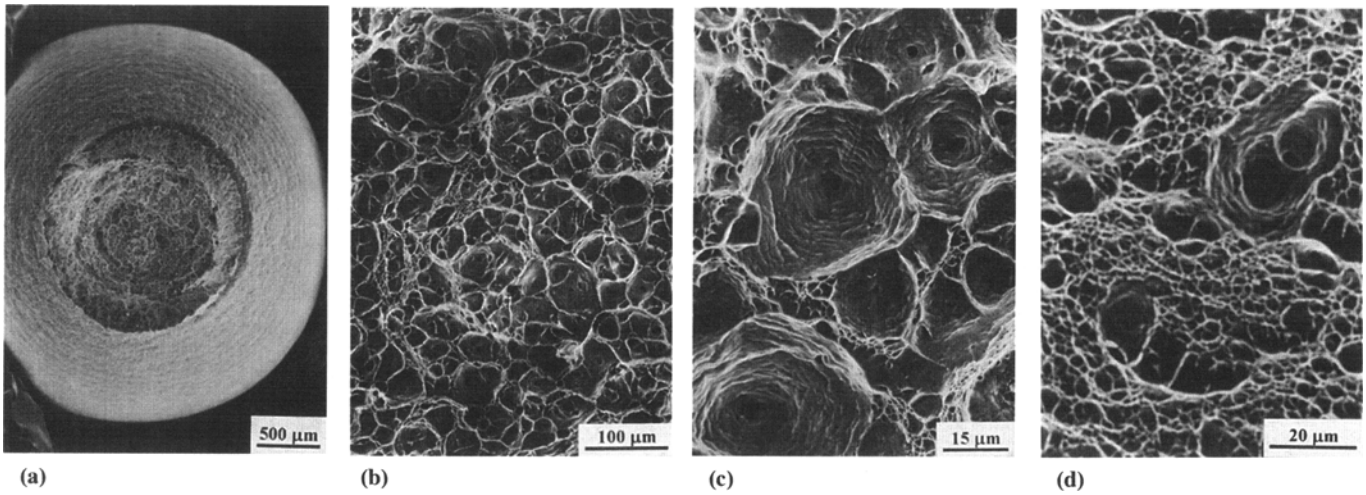


Fig. 13 SEM fractographs after homogenization and grain refinement

Table 6 Effect of heat treatment on mechanical properties

Condition	Ultimate tensile strength, MPa	Ultimate tensile strength, ksi	Elongation, %	Reduction in area, %
As received	1130	166	10.0	56
As received + 510 °C/3 h	2290	336	5.7	43
1200 °C/8 h + GR	1060	156	13.4	79
1200 °C/6 h + GR + 510 °C/3 h	2340	344	8.0	49
1200 °C/8 h + GR + 510 °C/3 h	2310	340	8.5	52

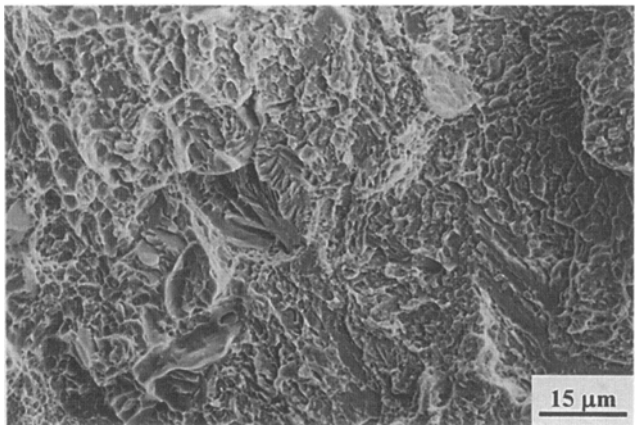
Table 7 Effect of heat treatment on impact strength

Sample condition	Impact energy, J
As received	120 ± 40
As received + 510 °C/3 h	6 ± 2
1200 °C/8 h + GR	170 ± 5
1200 °C/6 h + GR + 510 °C/3 h	14 ± 1
1200 °C/8 h + GR + 510 °C/3 h	20 ± 2

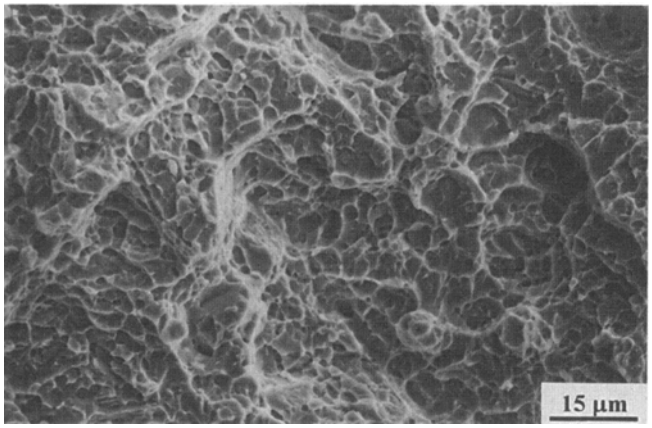
obtained in grain-refined specimens. The shear lip area is quite small compared to the as-received material. A higher-magnification image from the primary fracture area (Fig. 13b) shows a uniform distribution of dimples. The features within the dimples (Fig. 13c) indicate that substantial plastic flow occurred before the final rupture. The shear lip area exhibits both large and small dimples (Fig. 13d).

The impact strength of as-received and homogenized and grain-refined specimens was also measured (Table 7). The impact strength of the untreated material was on the order of 120 ± 40 and 6 ± 2 J in the annealed and aged conditions, respectively. The large fluctuations in values reflect the lack of homogeneity in the samples.

The data in Table 7 also indicate that toughness following homogenization and grain refinement improved substantially, especially when aged samples are compared. The difference can be of practical significance in critical engineering applica-



(a)



(b)

Fig. 14 Charpy fracture surfaces. (a) As-received and aged specimen. (b) Grain-refined and aged specimen

tions. The Charpy fracture surfaces obtained from the as-received and aged specimen (6 ± 2 J) and grain-refined and aged specimen (20 ± 2 J) are shown in Fig. 14. In the as-received condition (Fig. 14a), regions exhibiting a cleavage-type failure mode are present in addition to features that represent normal failure occurring by microvoid coalescence. In contrast, a uniform distribution of fine dimples typical of ductile fracture are visible in homogenized and grain-refined samples (Fig. 14b).

4. Summary

Segregation of alloying elements results in the formation of banded structure. If present above a certain critical level, segregation produces grain size variations across the bands. The banded structure can be reduced or eliminated by subjecting the material to a homogenization and grain refinement treatment, which also improves the mechanical properties of the material.

Acknowledgments

The authors thank Drs. A. ul Haq, A. Tauqir, and S.W. Husain for their valuable comments. Thanks are also due to Mr. Liaqat Ali and Mr. Afzal Hussain for their help in the mechanical testing and photography laboratories.

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