

Effect of Single and Double Austenitization Treatments on the Microstructure and Mechanical Properties of 16Cr-2Ni Steel

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Double austenitization (DA) treatment is found to yield the best combination of strength and toughness in both low-temperature as well as high-temperature tempered conditions as compared to single austenitization (SA) treatments. Obtaining the advantages of double austenitization (DA) to permit dissolution of alloy carbides without significant grain coarsening was attempted in AISI 431 type martensitic stainless steel. Structure-property correlation after low-temperature tempering (200 °C) as well as high-temperature double tempering (650 + 600 °C) was carried out for three austenitization treatments through SA at 1000 °C, SA at 1070 °C, and DA at 1070 + 1000 °C. While the increase in strength after DA treatment and low-temperature tempering at 200 °C is due to the increased amount of carbon in solution as a result of dissolution of alloy carbides during first austenitization, the increased toughness is attributable to the increased quantity of retained austenite. After double tempering (650 + 600 °C), strength and toughness are mainly found to depend on the precipitation and distribution of carbides in the microstructure and the grain size effect.

Keywords 431 stainless, double austenitization, martensitic steel, properties-stainless steel

1. Introduction

Martensitic stainless steel containing 16Cr-2Ni-0.15C, popularly known as AISI 431, has been a candidate material for a number of applications in the aerospace, marine, chemical, and food industries due to its excellent combination of high corrosion and oxidation resistance with good mechanical properties. Some applications of the steel include pump shafts, valve stems, high tensile fasteners, bomb racks, missile nozzle exhaust components, leather rollers, paper machine parts, and marine hardware.

The heat treatment methods suggested for 16Cr-2Ni stainless steel have been (a) austenitization between 950 and 1100 °C followed by quenching and (b) tempering between 200 and 300 °C for high strength, moderate toughness, and good corrosion resistance, with tempering between 600 and 700 °C for moderate strength, high toughness, and adequate corrosion resistance (Ref 1-5). The optimum combination of high strength and high toughness in the steel can be achieved only under carefully controlled heat treatment conditions.

Earlier study (Ref 6) on 16Cr-2Ni martensitic stainless steel revealed that austenitizing at a lower temperature of 1000 °C did not allow the large amount of alloy carbides to go into solution, leading to attainment of lower strength. On the other hand, a high austenitizing temperature of 1100 °C or above, however, helped in dissolution of alloy carbides and resulted in an increase in prior austenite grain size (besides increasing the δ -fer-

rite as well as retained austenite contents). The advantage of double austenitizing (DA) was proposed by Rao and Thomas (Ref 7, 8) and Sarikaya et al. (Ref 9) in Fe-4Cr-0.3C steels tempered at lower temperatures (200 to 265 °C). According to these authors, DA treatment permits dissolution of alloy carbides during the first austenitization treatment at a higher temperature, while the second low-temperature re-austenitization results in finer austenite grain size. Thus the DA treatment was attempted by earlier investigators to combine the benefits of achieving a homogeneous austenite phase that was free from undissolved alloy carbides and exhibited finer austenite grains.

Double austenitization treatment is expected to be beneficial in case of 16Cr-2Ni steel in controlling microstructural features so that an optimum combination of strength, toughness, and corrosion resistance is achieved. The present study has therefore been undertaken to evaluate the effects of single-austenitization (SA) and double-austenitization treatments on the microstructure and mechanical properties of a 16Cr-2Ni martensitic stainless steel, after tempering at a lower temperature (200 °C) as well as at a higher tempering temperature (double tempering at 650 + 600 °C).

2. Experimental Procedure

2.1 Material

The experimental steel was produced as a 30 kg heat in a vacuum induction furnace and cast as a cylindrical ingot of size 110 mm diameter by 345 mm height. The chemical composition of the steel is given in Table 1. The ingot was homogenized at 1150 °C for 14 h, radiographed, and cropped to remove the unsound portion. The sound portion of the ingot (110 mm diameter by 250 mm) was hot forged at 1100 °C to a billet of 30 mm thickness, which was hot rolled at 1100 °C to 16 mm thick plate.

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2.2 Heat Treatment

The 16 mm steel plate was given a stress relief annealing treatment for 2 h at 640 ± 5 °C followed by air cooling. Blanks of suitable size were cut along the direction of rolling for heat treatment experiments. Blanks were austenitized and tempered at different temperatures and times as given in Table 2.

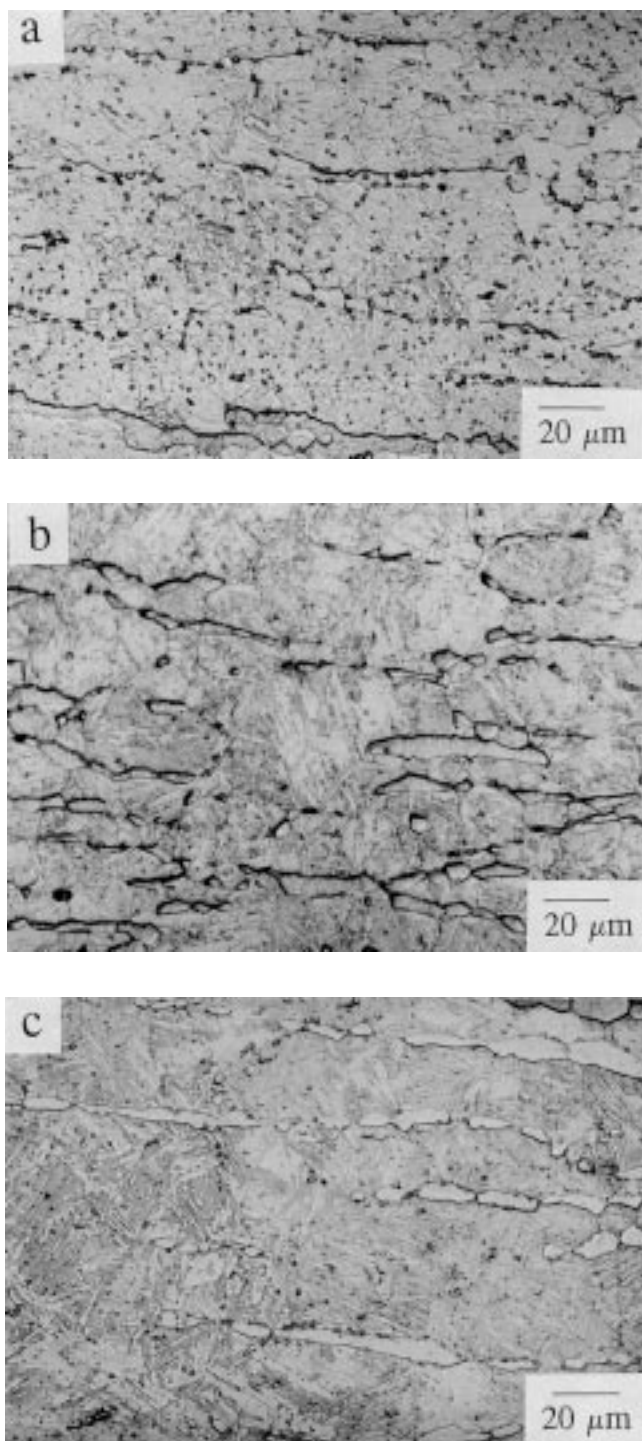


Fig. 1 Micrographs of the steel in as-quenched condition after austenitization at (a) 1000 °C, (b) 1070 °C, and (c) 1070 + 1000 °C

2.3 Mechanical Testing

Hardness was measured on the samples from all the heat treatment conditions at 30 kg load using a Vickers hardness tester. Room temperature tensile tests were performed on cylindrical specimens in the rolling direction with a gage diameter of 4.0 mm and a gage length of 25 mm at a strain rate 6.67×10^{-4} using an Instron test machine, model 1185. Room temperature Charpy V-notch (CVN) impact testing was carried out using a VEB Werkstoffprufmaschinen (Leipzig, Germany) machine on specimens of size 10 by 10 by 55 mm cut from the longitudinal direction. The notch was cut on the L-T direction as per ASTM E 23 (Ref 10). Both tensile and impact tests were performed on triplicate specimens.

2.4 Metallographic Techniques

Specimens for metallographic examination were prepared using conventional polishing techniques. The polished samples were etched using glyceric acid reagent (15 mL glycerol, 10 mL HCl, 5 mL HNO_3 , and 5 mL acetic acid) for revealing general microstructure and Villela's reagent was used to etch the prior austenite grain boundaries. The lineal intercept method was used to measure the grain size, and δ -ferrite in the optical microstructure was measured by point count method.

The fracture surfaces of tensile tested specimens were examined under scanning electron microscope (SEM), ISI 100 A at an accelerating voltage of 15 kV for fractography.

Thin foil specimens for examination under transmission electron microscope (TEM) were prepared from selected samples by electrolytic thinning at 25 mV using a 10% perchloric acid-90% glacial acetic acid electrolyte at 10 °C employing a Fischione twin jet electropolishing unit (Model 110-120, E.A. Fischione, INST. Mfg., Pittsburgh, PA). The thin foils were examined under a Phillips EM 430 TEM operating at 200 kV.

Retained austenite was measured on the heat treated specimens by the standard x-ray diffraction technique using molybdenum $\text{K}\alpha$ radiation as per ASTM E 975-84 (Ref 11).

3. Results

3.1 Optical Microscopy

The optical micrographs of the steel austenitized at different temperatures are shown in Fig. 1(a-c). These micrographs show stringers of δ -ferrite in a martensite matrix and undissolved carbides both along the δ -ferrite boundaries as well as within the martensite matrix. However, the amount of carbides decreased with increasing austenitization temperature from 1000 to 1070 °C. It was also observed that the DA treatment helped in taking most of the carbides into solution (Fig. 1c). The size and number of undissolved carbides were lower in the DA treated steel sample compared to the steel sample austenitized at 1000 °C (Fig. 1 a, c). However, some fine carbides were found to be present along the boundaries of δ -ferrite.

The optical micrographs showing prior austenite grain size of the steel samples that were given different austenitization treatments are shown in Fig. 2(a-c). The grain size increased from 15 to 30 μm with increase in austenitization temperature from 1000 to 1070 °C. The packet size of martensite laths was

also found to have increased marginally on raising the austenitizing temperature. The prior austenite grain size after the DA treatment was 22 μm .

The δ -ferrite content estimated by point count method on micrographs of samples treated at different austenitization temperatures was found to be constant at 8.0 vol%. Tempering did not have any effect on the δ -ferrite content of the steel.

The retained austenite content increased from 7.9% when austenitized at 1000 °C to 12.5% at 1070 °C. On DA treatment the retained austenite content of the steel further increased to 14.2%. No significant change in the volume fraction of retained austenite was noticed on tempering the samples at 200 °C as compared to the as-quenched condition. However, the retained austenite reduced to less than 2.0 vol% when the steel was given a double tempering treatment (650 + 600 °C) after different austenitization treatments.

The variation of grain size and retained austenite contents of the steel for different austenitization treatments is given in Table 3.

3.2 Transmission Electron Microscopy

The TEM of the as-quenched samples of the steel austenitized at 1000 °C, austenitized at 1070 °C, and double austenitized (1070 + 1000 °C) are shown in Fig. 3(a-c) respectively. The lath width remained almost the same in all the three samples (SA-1, SA-2, and DA). Samples in both

the conditions (namely, as quenched from different austenitizing temperatures, as well as after tempering at 200 °C for 1 h) showed typically lath martensite and interlath contiguous films of retained austenite. Bright field and dark field images of the DA treated samples that were subsequently tempered at 200 °C are shown (Fig. 4a, b) respectively. The selected area diffraction pattern (SADP) from an austenite reflection and its schematic representation are shown in Fig. 4(c, d) respectively.

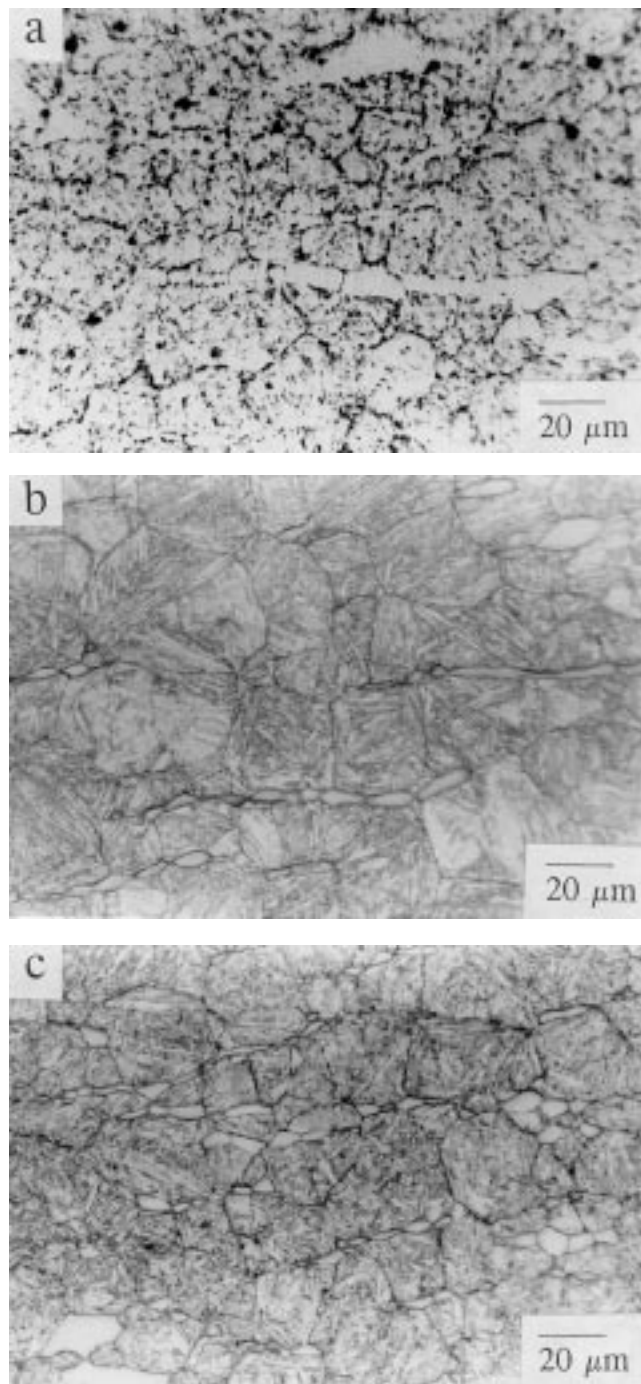


Fig. 2 Micrographs showing prior austenite grain size of the steel austenitized at (a) 1000 °C, (b) 1070 °C, and (c) 1070 + 1000 °C

Table 1 Steel composition

Element	Composition, wt%
C	0.18
Si	0.77
Mn	0.17
Cr	16.1
Ni	3.0
S	0.003
P	0.015

Table 2 Heat treatment

Sample	Austenitization
SA-1	1000 °C, oil quenched 30 min
SA-2	1070 °C, oil quenched 30 min
DA	1070 °C, oil quenched 20 min +1000 °C, oil quenched 30 min

Note: All samples were tempered at low temperature (200 °C and air cooled 1 h) and double tempered at high temperature (650 °C and air cooled 1 h + 600 °C and air cooled 1 h).

Table 3 Variation in retained austenite and grain size as a function of heat treatment

Sample	Retained austenite, vol%			Prior austenite grain size, mm
	As quenched	After tempered at:		
		200 °C	650 + 600 °C	
SA-1	7.9	7.5	<2.0	15
SA-2	12.5	12.0	<2.0	30
DA	14.2	13.9	<2.0	22

The double tempered (650 + 600 °C) steels after the three-austenitization treatment showed the recovered lath structure with well developed subgrains and low dislocation densities within subgrains. The thin foils showed finely precipitated interlath carbides in the double tempered sample of SA-1 whereas interlath and intralath carbides were seen in DA treated steel samples. The carbides were identified by selected area diffraction as $M_{23}C_6$ carbides. The TEMs of double tempered samples of SA-1 and DA treated steel are shown in Fig. 5(a-d) and Fig. 6(a-c) respectively.

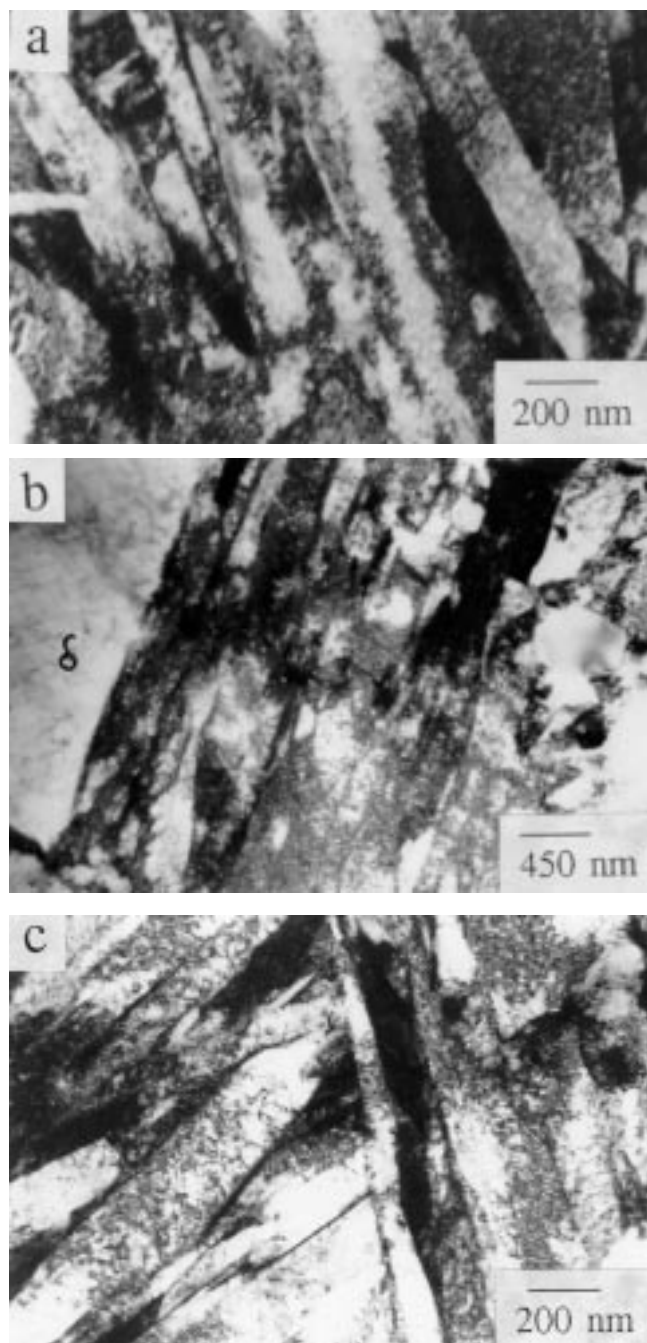


Fig. 3 TEM micrographs of as-quenched steel austenitized at (a) 1000 °C, (b) 1070 °C, and (c) 1070 + 1000 °C. Dislocated lath martensite structure is shown.

3.3 Mechanical Properties

On increasing the austenitization temperature from 1000 to 1070 °C, the as-quenched hardness increased from 442 to 468 HV. The hardness attained by DA treatment was similar to that of 1000 °C austenitized condition. Variations in hardness on tempered samples quenched from different austenitization treatments are given in Table 4. The hardness did not change significantly on tempering at 200 °C for 1 h. However, on double tempering at 650 + 600 °C for 1 h at each temperature, the increase of hardness was from 264 to 301 HV, corresponding to increase in austenitization temperature from 1000 to 1070 °C. For the same tempering treatment the DA treatment gave a hardness which was 18 HV more than the sample austenitized at 1000 °C, which was unlike its behavior either in as-quenched or 200 °C tempered conditions.

Variation in tensile properties is given in Table 4 for different austenitization treatments followed by tempering at 200 °C and double tempering at 650 + 600 °C. Both yield strength and tensile strength were the highest in the sample austenitized at 1070 °C and the lowest in the samples austenitized at 1000 °C. Strength levels of the DA treated sample fell between the two. The ductility parameters did not show significant variation due to changes in the austenitization treatments.

The Charpy impact energy data at room temperature for different austenitization treatments are given in Table 4. The impact energy was higher for both the 200 °C tempered as well as 650 + 600 °C double tempered conditions, which were austenitized at 1000 °C (compared to 1070 °C). The impact energy of the DA treated sample showed a considerable improvement over that obtained by either of the two single austenitization treatments in both the tempered conditions. The impact toughness after double tempering was vastly superior to that of 200 °C tempering for all austenitization treatments.

3.4 Fractography

The SEM fractographs of tensile specimens fractured at room temperature in respect of hardened and double tempered samples SA-1, SA-2, and DA are shown (Fig. 7 a-c). The fracture surfaces of all the three samples showed characteristic dimpled rupture. However, the dimple size was found to vary considerably, showing large dimples in the sample austenitized at 1000 °C and much finer dimples in the sample austenitized at 1070 °C. The fracture surface of the tempered sample after DA treatment showed intermediate size and fine dimples. The SEM fractographs of impact specimens for corresponding heat treatments were more or less similar to the tensile specimens.

4. Discussion

The experimental results of single and double austenitization treatments on quenched and tempered 16Cr-2Ni stainless steel indicate the following behavior:

- During conventional single austenitization treatments, the carbide dissolution as well as the grain size increases with an increase in austenitization temperature.
- The double austenitization treatment promotes carbide dissolution with insignificant increase in grain size.

- Judicious selection of austenitization temperature (single or double) helps maintain a constant volume fraction of δ -ferrite.
- The volume fraction of retained austenite increases with increase in single austenitization temperature and also further increases with the DA treatment.
- The retained austenite remains stable on tempering the steel at 200 °C.
- The retained austenite gets almost completely transformed by the double tempering treatment at 650 + 600 °C.
- Hardness and tensile strength of the steel increase with increase in single austenitization temperature from 1000 to

1070 °C, while after DA treatment the corresponding properties lie between those achieved by lower (1000 °C) and higher (1070 °C) single austenitization treatments.

- The single austenitization treatment (at the lower or higher temperature) or the DA treatment does not affect significantly the tensile ductility in the tempered condition.
- Room temperature Charpy impact toughness of the steel after tempering (either at low or high temperature) reduces marginally with increasing austenitization temperature of SA treatment. However, on DA treatment, the toughness improves.

Table 4 Variation in mechanical properties as a function of heat treatment

Sample	Tempering temperature, °C	Hardness, HV	0.2% yield strength, MPa	Ultimate tensile strength, MPa	Elongation, %	Reduction in area, %	Charpy V notch impact energy, J
SA-1	200	427	1029	1373	14	45	47
	650 + 600	264	673	834	18	58	94
SA-2	200	458	1080	1645	13	40	43
	650 + 600	301	812	960	16	55	82
DA	200	433	1067	1411	15	50	58
	650 + 600	282	774	943	19	60	110

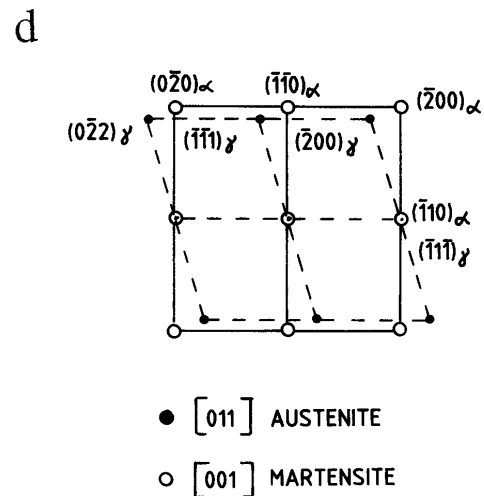
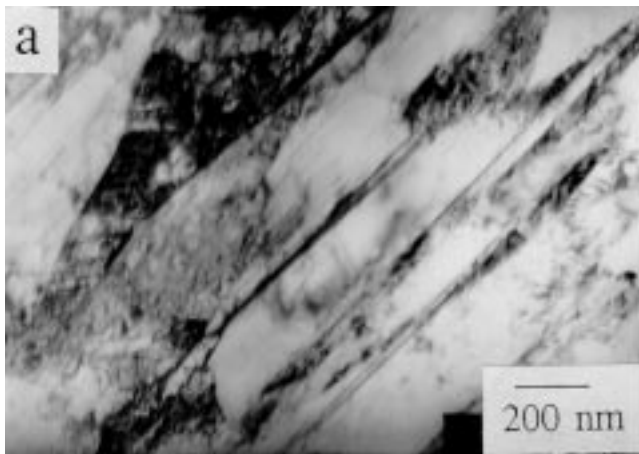
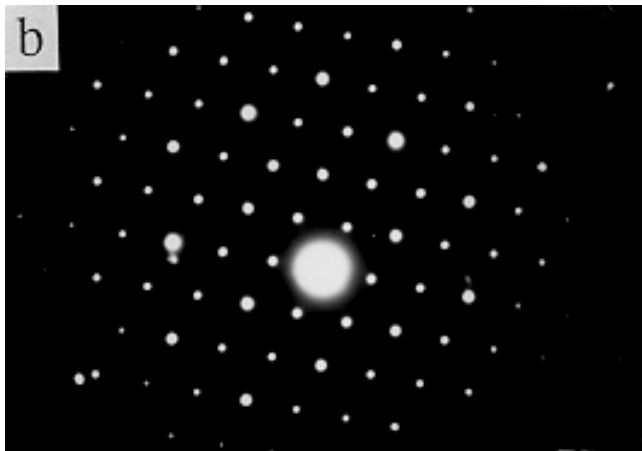
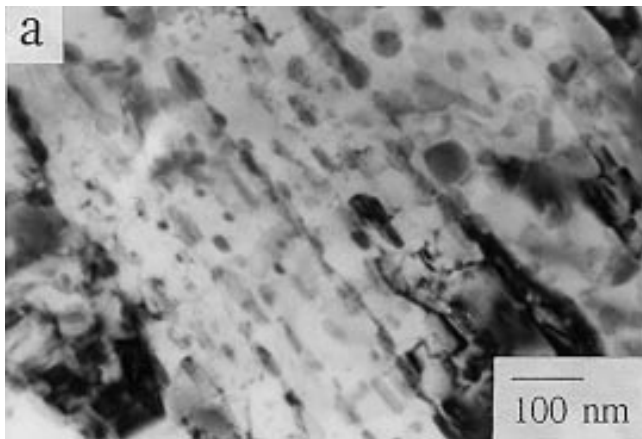


Fig. 4 TEM micrographs of double austenitized (DA) treated and 200 °C tempered steel showing dislocated lath martensite and interlath films of retained austenite. (a) Bright-field (BF) image. (b) Dark-field (DF) image. (c) Selected area diffraction pattern (SADP) from lath boundary austenite. (d) Schematic representation of (c)



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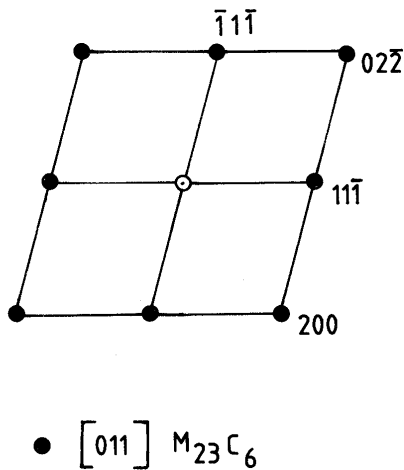


Fig. 6 TEM micrographs of the DA treated (1070 + 1000 °C) and double tempered (650 + 600 °C) steel showing interlath and intralath carbides and recovered martensite. (a) BF image. (b) SADP of intralath $M_{23}C_6$ carbide. (c) Schematic representation of (b)

Very low dislocation density and well-developed subgrains after double tempering the steel suggest that only recovery (and not recrystallization of lath structure) occurs after such treatment. The double tempering treatment results in almost complete transformation of retained austenite as evidenced by

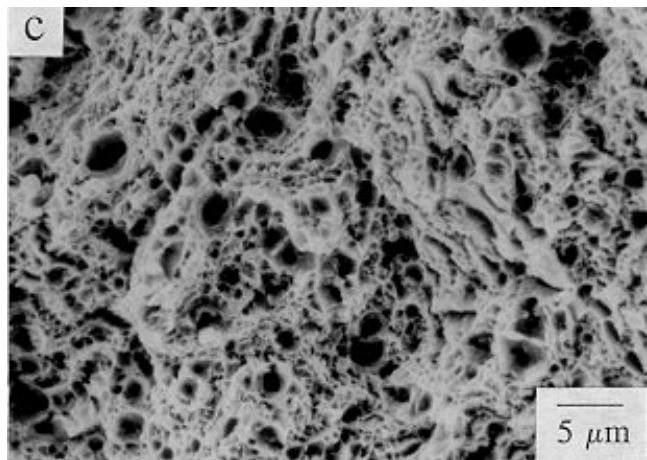
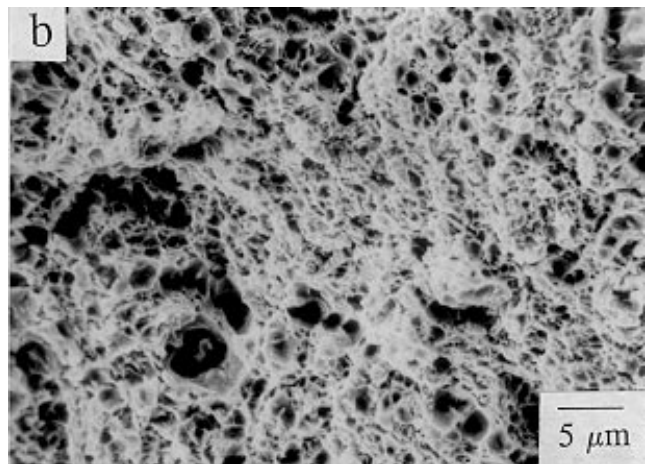
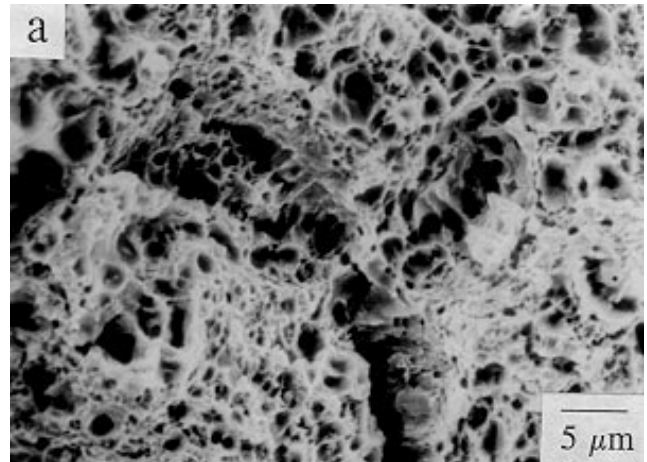


Fig. 7 SEM fractographs of fracture surfaces of tensile test specimens showing variation in dimpled rupture in hardened and double tempered samples. (a) SA-1. (b) SA-2. (c) DA

x-ray diffraction data irrespective of its amount when quenched from either low-temperature or high-temperature SA or after DA treatment. Reduction of retained austenite from 12 to 14% to less than 2% following double tempering suggests that due to carbide precipitation during first tempering, the austenite

leaner in carbon gets transformed to martensite, and on second tempering at 600 °C, the martensite formed after first tempering transforms to carbides and ferrite. δ -ferrite content does not change on tempering the steel up to 650 °C. The high chromium content in the δ -ferrite causes chemical stability of the phase and therefore, the short periods of tempering do not result in its decomposition. L. Ning et al. (Ref 5) in studies on similar steel have observed the δ -ferrite to remain undecomposed up to 700 °C tempering.

Double tempering (650 + 600 °C) of steel after SA-1 causes predominantly interlath carbide precipitation, which forms due to transformation of interlath retained austenite. Martensite being leaner in carbon content does not permit higher amounts of intralath carbide precipitation following double tempering in this sample. On the other hand, copious precipitation of $M_{23}C_6$ carbides in the interlath as well as the intralath regions is due to carbon-enriched martensite in both the SA-2 and the DA treated samples. Earlier investigations (Ref 16, 17, 18, 19) have reported the precipitation of $M_{23}C_6$ type carbides in 12% Cr and 16% Cr martensitic stainless steels when tempered between 600 and 700 °C.

4.2 Mechanical Properties

The increase in the as-quenched hardness with increasing austenitization temperature is because of the increase in carbon content of the austenite transforming to martensite on quenching. The marginal decrease in the as-quenched hardness of DA treated samples (compared to that of SA-2) could be attributed to the slightly higher retained austenite content of the DA treated samples and the decrease in carbon content due to carbide precipitation during second-stage austenitization. The marginal decrease in hardness for all the austenitization conditions after tempering at 200 °C probably is due to onset of recovery of martensite and consequent reduction in dislocation density. The present investigation demonstrates that the DA treatment results in yield strength (YS) and ultimate tensile strength (UTS) intermediate between the two single austenitization treatments (1000 °C and 1070 °C) with marginally superior tensile ductilities and notch toughness values when the steels are tempered at 200 °C because of (a) austenite grain size being intermediate between the SA-1 and SA-2 treatments, (b) slightly increased amount of retained austenite, and (c) intermediate undissolved carbide content. The δ -ferrite content has not significantly changed due to the different austenitization treatments. The synergistic effects of the microstructural features in DA treated steel are responsible for the improvement in impact toughness in the low temperature (200 °C) tempered condition compared to either of the single austenitization treatments.

In a low alloy steel containing 0.3% C, 4.0% Cr, and 2.0% Mn, Rao and Thomas (Ref 8) observed that in 200 °C tempered steel DA treatment (1100 + 870 °C) resulted in a gain of 13 J in impact toughness against a loss of 75 MPa in yield strength as compared to single austenitization at 1100 °C. In another steel containing 0.3% C, 4.0% Cr, and 5.0% Ni, CVN energy was unaffected by DA treatment followed by tempering at 200 °C, as compared to single austenitization at 1100 °C. However, the DA treatment was found to have resulted in a better combination of strength and impact toughness.

In another investigation by Sarikaya et al. (Ref 9), low alloy steels of base composition 0.25% C, 3.0% Cr with either 2% Mn or 2% Ni exhibited significant grain refinement while retained austenite remained unaffected following DA treatment (1100 + 900 °C) as compared to single austenitization (SA) at 1100 °C. They found that in 200 °C temper condition following DA treatment, the slight benefit (2 to 4 J) in Charpy impact energy has been achieved with a considerable loss of YS (about 75 to 100 MPa) as compared to the SA treatment.

In the double tempered (650 + 600 °C) condition, the microstructural variables that would affect the mechanical properties in differently austenitized conditions are limited to the volume fraction of undissolved and reprecipitated carbides and the grain size. The effect of δ -ferrite is discounted due to constant volume fraction in differently austenitized and double tempered condition, while retained austenite dips to the same low level after double tempering in all the samples irrespective of the austenite content in as quenched condition, thus causing negligible effects on the properties.

The higher yield strength and ultimate tensile strength of the SA-2 treated steel as compared to SA-1 or DA treated steel after double (650 + 600 °C) tempering is attributable to increased precipitation strengthening due to precipitation of larger number of finer carbides. The relatively lower strength of SA-1 is because the increase in strength due to the finer grain/packet size of sample is not found to compensate for the decrease in strength caused by the lower carbide precipitation after double tempering.

The larger dimples in the tensile fracture specimen of sample SA-1 (as compared to those in sample SA-2) indicate void nucleation at coarse undissolved carbides. Finer dimples in sample SA-2 indicate that void nucleation has occurred at fine precipitated carbides. Large particles nucleate voids at lower stresses (Ref 20), and finer precipitated carbides impart resistance to void nucleation, thereby withstanding higher stresses (Ref 21).

The higher toughness of sample SA-1 as compared to sample SA-2 could be attributed to softer martensite and finer grain size.

With the formation of void sheets due to finer carbides between coarse undissolved carbides being less in sample SA-1, void growth is retarded, resulting in better toughness as compared to sample SA-2 (Ref 21). Void growth and coalescence promoted by finer precipitated carbides together with higher grain size contribute to lower toughness of sample SA-2.

The improvement in impact toughness of the DA treated sample in double tempered condition as compared to low and high single austenitized samples could be attributed to the finer grain size (as compared to SA-2) and lower content of undissolved carbides (as compared to SA-1). This results in higher resistance to void nucleation (as compared to sample SA-1) and higher resistance to void sheet formation (as compared to sample SA-2).

In a study by Chang et al. (Ref 22) with 3Ni-1Cr-0.4Mo-0.1V-0.34C steel, virtually no change was reported in grain size and tensile properties (YS, TS, percentage elongation, and reduction area) following the DA treatment (1000 + 840 °C) followed by tempering at 570 °C as compared to the single austenitization at 840 °C followed by tempering at 600 °C. However, the study has shown an improvement of 16 J in CVN energy after DA treatment followed by tempering.

Thus earlier investigations on low alloy steels show only a minor benefit of double austenitization treatment, which does not really justify recommending the treatment for industrial applications, as the benefit in the strength + toughness combination after DA treatment might not compensate for the cost of energy consumption in carrying out the DA treatment.

The present investigation reveals that double austenitization (1070 + 1000 °C) improves the impact toughness of 16Cr-2Ni steel at a small expense of YS and TS as compared to single austenitization at high temperature (1070 °C). On the other hand, the DA treatment is found to improve impact toughness as well as YS and TS considerably as compared to the specified values (Ref 2) following single austenitization at lower temperature (1000 °C). Due to fewer undissolved carbides in the steel, the DA treatment is also expected to improve corrosion resistance as compared to 1000 °C single austenitization treatment. The DA treatment is also found to provide a better combination of strength and toughness as compared to either of the single austenitization treatments.

5. Conclusions

The following conclusions can be drawn based on the present investigation:

- Undissolved carbides decrease significantly following high temperature (1070 °C) single austenitization as compared to low temperature (1000 °C) single austenitization treatment. Few undissolved carbides remained in the steel after DA (1070 + 1000 °C) treatment.
- The δ -ferrite is practically unaffected by any of the austenitization treatments.
- Retained austenite content increases following single austenitization at 1070 °C as compared to 1000 °C, while DA treatment further increases the retained austenite content.
- The grain size increases with increase in single austenitization temperature from 1000 to 1070 °C, whereas DA treatment results in an intermediate grain size.
- The amount of precipitated carbides is lower in single low-temperature austenitized steels as compared to single high-temperature austenitized or DA steel in which the precipitated carbides get uniformly distributed in the microstructure.
- With increase in single austenitization temperature from 1000 to 1070 °C, the yield strength and ultimate tensile strength of both low temperature (200 °C) as well as high temperature (650 + 600 °C) tempered steel increases. The corresponding properties of DA treated steel were higher as compared to 1000 °C steel that has received SA treatment.
- There has been no significant effect on the tensile ductility following any austenitization treatment.
- Charpy impact toughness decreases with increase in single austenitization temperature from 1000 to 1070 °C, whereas DA treatment results in significant improvement in impact toughness as compared to both single austenitization treatments.

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