Effect of Thermo-mechanical Control Process on Strengthening of a 22Mn-13Cr-5Ni Austenitic Stainless Steel Plate for Cryogenic Use

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The effect of thermo-mechanical control process (TMCP) on strengthening of a 22Mn-13Cr-5Ni austenitic stainless steel plate for cryogenic use has been investigated by using an intermittent hot compression test. Finish-compression temperature, cooling rate and start-cooling temperature are the factors controlling the recrystallized austenite grain size. The austenite grain becomes finer as the finish-compression temperature decreases and as the cooling rate and the start-cooling temperature increase in the recrystallization region, all of which are effective for strengthening the steel. On the basis of these results, a steel plate of thickness 70 mm was produced on a large scale under the optimum TMCP conditions and the mechanical properties of the steel plate were investigated. The TMCP steel attained a higher 0.2 % yield strength than that of the solution heat-treated (ST) steel at -269°C , though the fracture toughness was slightly reduced.

KEY WORDS: thermo-mechanical control process; high manganese austenitic stainless steel; recrystallization; grain refinement; strength.

1. Introduction

The industrial field related to magnetism including nuclear fusion reactors and the Mag-lev transportation system, *etc.*, is in the experimental stage for practical application. High strength non-magnetic austenitic steels are required as the structural materials for such equipment. Solid-solution hardening^{1,2)} using elements such as chromium, molybdenum and nitrogen, precipitation hardening^{3,4)} using vanadium carbide and nitride, and work hardening⁵⁾ have been generally applied to strengthening of the austenitic steels. Strengthening by the grain refinement has been widely investigated in the field of ferritic steels by applying a thermo-mechanical control process (TMCP).⁶⁾ However, there are few studies on strengthening by grain refinement in the field of austenitic steels.^{6,7)}

In this study, the effect of TMCP on strengthening austenitic steel was investigated using a 22Mn-13Cr-5Ni steel for cryogenic use (22Mn steel), and the discussion was restricted to the recrystallized structure which has high ductility and toughness. First, the effect of TMCP on hardness and microstructure in the 22Mn steel was systematically investigated by an intermittent hot compression test. The effective use of TMCP as a method of strengthening was discussed from a metallurgical viewpoint. Secondly, a 22Mn steel plate of thickness 70 mm was produced under selected TMCP conditions in a large scale mill. The mechanical properties of the steel plate were investigated and the efficacy of TMCP was discussed.

2. Experimental Procedures

The material used was a high-manganese austenitic stainless steel for cryogenic use. The chemical composition is shown in Table 1. The steel ingot melted in a 15 t electric furnace was hot-rolled to a 200-mm-thick slab. Cylindrically shaped specimens 8 mm in diameter and 12 mm in height were machined from the quarter thickness of the slab in the perpendicular direction. For simulating the actual hot-rolling process, the specimens were intermittently hot-deformed with uniaxial compression using a hot deformation simulator.

Fig. 1(a) shows the schematic diagram for the simulated condition, which almost corresponds to that of the actual plate rolling from 200-mm-thick slab to 70-mm-thick plate. The specimens were first reheated to 1200°C for 5 min, followed by hot-compression from 12 to 4.2 mm in 7 passes at the cooling rate of 10°C/s . The strain rate and total reduction were 10 s^{-1} and 65 %, respectively. The effects of the finish-compression temperature, cooling rate, start-cooling temperature on strengthening were investigated as important process parameters of TMCP. The finish-compression temperature was changed in the range of temperature from 800 to 1000°C . The average cooling rate from the finish-compression tem-

Table 1. Chemical composition of steel tested.

(wt% in check analysis)							
C	Si	Mn	P	S	Ni	Cr	N
0.04	0.34	21.82	0.013	0.004	4.94	12.84	0.23

perature below 150°C varied from 0.17 to 30°C/s. The cooling rate of 0.17°C/s corresponds to that of as-rolled steel plate with a thickness of 70 mm. The starting temperature of accelerated-cooling varied from 700 to 1 000°C.

The behavior of recrystallization was investigated under the hot-compression and annealing schedule represented in Fig. 1(b). The specimens were first reheated to 1 200°C for 5 min, followed by hot-compression from 12 to 8.4 mm in 1 pass. The strain rate and total reduction were 10 s⁻¹ and 30 %, respectively. The compression and holding temperature was changed from 900 to 1 100°C. After deformation, the specimens were held isothermally at the same temperature. The holding time varied at intervals between 0.1 and 100 s. The average cooling rate was 30°C/s owing to observing the change of the austenite structure. A solution heat-treated (ST) specimen at 1 050°C for 5 min was also prepared to simulate the conventional manufacturing process.

The microstructures of all specimens were observed using an optical microscope after etching with aqua regia at the center of the plane parallel to the longitudinal direction. The austenite grain size was measured by using the circular diameter with an image analyzer. The Vickers hardness (HV 10 kgf) was measured at the same position as the microstructural observation in accordance with JIS Z2244. Vickers hardness was used as a factor of strength, because the

specimens were too small to undergo tension test. A good correlation between Vickers hardness and 0.2 % yield or tensile strength was reported by the authors on a 25Mn austenitic steel.⁹⁾ The substructures were also observed using a transmission electron microscope (TEM).

Specimens for the tensile test were cylindrical samples of size 7 mm ϕ ×50 mm (at a reduced section) taken perpendicular to the rolling direction from the quarter thickness of the plate. A 10 t cryogenic tensile testing machine¹⁰⁾ was used for the tension test, which was conducted at -269°C in liquid helium. Compact tension specimens 25 mm thick taken from the quarter thickness of the plate in the *T-L* orientation were used for the fracture toughness test, which was conducted at -269°C in liquid helium by means of the unloading compliance method which was described in ASTM E813-81 standards.¹¹⁾

3. Results and Discussion

3.1. Effect of Finish-compression Temperature on Strengthening

The effect of the finish-compression temperature on hardness and austenite grain size is shown in Fig. 2, and the microstructural change with the finish-compression temperature is shown in Fig. 3. In this case, the steels were hot-compressed at the range of finish-compression temperature from 800 to 1 000°C, and then cooled at 0.17°C/s immediately after deformation.

Austenite grains become finer and hardness in-

Fig. 1. Schematic diagrams of test condition.

Fig. 2. Effect of finish-compression temperature on hardness and austenite grain size.

Fig. 3. Microstructural change with finish-compression temperature.

creases as temperature decreases. A rapid increase in hardness takes place at the range of temperature from 900 to 800°C. Though the hardness at 1 000°C is almost the same as that of the ST steel at 1 050°C, the hardness below 950°C is higher than that of the ST steel. Steels finish-compressed over 900°C give rise to a recrystallized structure, while steel finish-compressed at 800°C exhibits a non-recrystallized structure as shown in Fig. 3. In the recrystallization region, austenite grains become finer and hardness increases as temperature decreases. Therefore, it is possible to use the effect of grain refinement by hot-deformation on strengthening. On the other hand, though a rapid increase in hardness is observed in the non-recrystallization region, it is difficult to apply it to strengthening because it would be thought toughness decreased. Therefore, the effect of TMCP on strengthening is discussed in the region where the finish-compression temperature is over 900°C in the following sections.

3.2. Effect of Cooling Rate on Strengthening

Fig. 4 shows the effect of the cooling rate on hardness and austenite grain size after hot-deformation. In this case, the steels were hot-compressed at the finish-compression temperature of 1 000°C, and then cooled from 0.17 to 30°C/s immediately after deformation. The austenite grains become finer and hardness increases as the cooling rate increases. It should be noted here that a rapid increase in hardness is observed at the range of cooling rate from 3 to 10°C/s, although hardness increases slowly at a cooling range below 3°C/s.

Fig. 5 shows the microstructural and substructural changes with cooling range. The steels cooled at 0.17 and 3°C/s exhibit a recrystallized structure with low dislocation density, which is almost the same as that of ST steel. The austenite grains at the cooling rate of 0.17°C/s are coarser than those cooled at 3°C/s. This fact shows that the accelerated-cooling acts as a restraint factor of austenite grain growth. On the other hand, the steel cooled at 30°C/s has finer grains than that of the steels cooled at 0.17 and 3°C/s, and includes partially-recrystallized structure mixed with the recrystallized structure showing low dislocation density and the non-recrystallized structure showing

Fig. 4. Effect of cooling rate on hardness and austenite grain size.

high dislocation density. Therefore, the rapid increase in hardness described above can be attributed to such a partially-recrystallized structure. Recrystallization is restrained when the cooling rate is higher than 10°C/s.

It is found, from the results above mentioned, that the cooling rate in TMCP divides an austenite structure into a recrystallized, non- and partially-recrystallized structure, and is therefore one of the factors determining the austenite structure. Furthermore, in the recrystallization region, it is found that the cooling rate acts as a factor affecting the austenite grain size. In the case based on such discussion, the lower limit of the finish-rolling temperature where a recrystallized structure can be obtained, should be changed by cooling rate.

Fig. 6 represents the effect of the finish-compression temperature on austenite grain sizes and hardness. In this case, the cooling rate was varied from 0.17 to

Fig. 5. Microstructural and substructural changes with cooling rate.

Fig. 6. Effect of finish-compression temperature and cooling rate on hardness and austenite grain size.

Fig. 7. Microstructural change with finish-compression temperature and cooling rate.

means of selecting the finish-compression temperature and cooling rate. Fig. 7 shows the conditions of the finish-compression temperature and the cooling rate to obtain recrystallized structure. It is needless to say that it would be better to select a condition near the lower limit of the recrystallization region.

3.3. Effect of Start-cooling Temperature on Strengthening

Fig. 8 shows the effect of the start-cooling temperature on hardness and austenite grain size. In this case, the steels were hot-compressed at the finish-compression temperature of 1000°C, and cooled to the several appointed temperatures at 0.17°C/s, and then accelerated-cooled below 150°C at 3°C/s. As the start-cooling temperature decreases, that is to say, as the air cooling time till start-accelerated-cooling increases, austenite grain becomes coarser and hardness decreases. However, hardness scarcely changes and is almost the same as that of ST steel at the

30°C/s. The lower limits of the finish-compression temperature where a recrystallized structure can be obtained, are 900 and 950°C at the cooling rates of 0.17 and 3°C/s, respectively. It is also found that austenite grains can be refined up to about 10 μm by

start-cooling temperature below 900°C.

This fact shows that austenite grain size is varied by start-cooling temperature and hardness is consequently controlled. As for the reason why hardness decreases with decreasing start-cooling temperature, it would be thought that fraction recrystallized increased or recrystallized austenite grain coarsened with time.

Fig. 9 shows the microstructural change with start-cooling temperature. All of them exhibit recrystallized structure. The recrystallized grains become coarser as the start-cooling temperature decreases. Therefore, the decrease in hardness during air-cooling is due to the growth of recrystallized austenite grain.

The result described above was obtained in the case where the finish-compression temperature was 1 000°C. A more detailed investigation was conducted on the behavior of recrystallization immediately after hot-compression.

Fig. 8. Effect of start-cooling temperature on hardness and austenite grain size.

Fig. 9. Microstructural change with start-cooling temperature.

Fig. 10 shows the changes in hardness and in fraction recrystallized with holding temperature and time after hot-compression with 1 pass. In this case, compression temperature was varied from 900 to 1 100°C. The steels have almost recrystallized after holding for 10 s at 1 000 and 1 100°C. On the other hand, it needs over 100 s holding to obtain recrystallized structure at 900°C.

Fig. 11 shows the microstructural change with holding time after hot-deformation. In the case where hot-compression temperature is 1 000°C, the steel has almost recrystallized after 10 s holding, and the recrystallized grain growth starts after more than 10 s holding. On the other hand, recrystallization does not finish completely even after 100 s holding at 900°C.

This fact suggests that the suitable start-cooling temperature should be chosen in accordance with the finish-rolling temperature when TMCP is applied to the production. That is to say, accelerated-cooling immediately after hot-rolling is necessary to restrain the recrystallized austenite grain growth in cases of a high finish-rolling temperature such as 1 000 and

Fig. 10. Change in hardness and fraction recrystallized with holding temperature and time after hot-deformation.

Fig. 11. Microstructural change with holding time after hot-deformation.

1 100°C. On the other hand, an appropriate interval for air-cooling before accelerated-cooling is necessary to obtain the recrystallized structure in case of a comparatively low finish-rolling temperature of 900°C.

3.4. Metallurgical Role of TMCP on Strengthening

So far, the effect of the finish-rolling temperature, the cooling rate and the start-cooling temperature on hardness and austenite grain size has been investigated. From the results, it was found that these parameters act as the factors determining the austenite microstructure.

Fig. 12 shows the relationship between hardness and austenite grain size. In the recrystallization region, a good correlation exists between hardness and austenite grain size which is not affected by the manufacturing processes, that is, accelerated-cooling, air-cooling and solution heat-treatment. Hardness increases as the austenite grain becomes finer.

Austenite grain can be refined from 20 to 10 μm by the application of TMCP to the 22Mn steel. Consequently, hardness of the 22Mn steel can also increase from 160 to 190 in HV in the recrystallization region. In the non- or partially-recrystallization region, the steels have a higher hardness than 210 in HV. However, the use of the non- or partially-recrystallized structure for strengthening is not suitable, because it would be thought these structures caused a deterioration in ductility and toughness of the steel for cryogenic use.

Fig. 13 shows schematic diagrams summarizing the present results on the effect of TMCP. Recrystallized austenite grain becomes finer as the finish-rolling temperature decreases, cooling rate and start-cooling temperature increase, and can be refined up to 10 μm under optimum TMCP conditions. In this case,

Fig. 12. Relationship between hardness and austenite grain size.

the finish-rolling temperature plays a part in refining the austenite grain, and the cooling rate and the start-cooling temperature play parts in restraining the recrystallized austenite grain growth.

3.5. Mechanical Properties of TMCP Steel

The 22Mn steel plate of thickness 70 mm, produced on a large scale under the optimum conditions dis-

cussed above, was tested for tensile properties at room temperature and -269°C , and fracture toughness at -269°C . The steel was first reheated to 1200°C and then hot-rolled from 200-mm-thick slab to 70-mm-thick plate. In this case, the finish-rolling temperature was 950°C . After hot-rolling, the steel plate was air-cooled for 50 s and then accelerated-cooled below 150°C at the average cooling rate of 5°C/s . As a comparative material, a steel plate solution heat-treated at 1050°C for 140 min was also prepared.

Table 2 shows the mechanical properties of the TMCP steel and the ST steel at room temperature and -269°C .

The hardness of the TMCP steel, is 189 in HV at room temperature, is 23 in HV higher than that of ST one. The result almost accords with that of the simulation test described earlier.

The 0.2 % yield and tensile strengths for the TMCP steel are 358 and 678 MPa at room temperature, and are 1240 and 1659 MPa at -269°C , respectively. Both the 0.2 % yield and tensile strengths of the TMCP steel are about 50 MPa higher than those of the ST steel at room temperature, and are about 100 MPa higher than those at -269°C .

The fracture toughnesses for the TMCP steel and the ST steel are 194 and 213 $\text{MPa}\sqrt{\text{m}}$ at -269°C , respectively. The former is slightly reduced in comparison with the latter.

Fig. 14 shows the relationships between 0.2 % yield strength and fracture toughness at -269°C of 22Mn and AISI Type 304N steels.¹²⁾ The 22Mn

steels have a good balance of 0.2 % yield strength and fracture toughness superior to that of the AISI Type 304N steels. The 22Mn steel produced by TMCP attains a higher 0.2 % yield strength, though the fracture toughness becomes a little lower than that of the ST steel.

Fig. 14. Relationships between 0.2 % YS and K_{IC} of 22Mn-13Cr-15Ni and AISI Type 304N steels at -269°C .

Fig. 13. Schematic diagrams summarizing results on effect of TMCP.

Table 2. Mechanical properties of TMCP and ST type steels.

Heat treatment	Test temp. ($^{\circ}\text{C}$)	Tensile properties			Fracture toughness K_{IC} ($\text{MPa}\sqrt{\text{m}}$)	Hardness HV (10 kgf)
		0.2 % YS (MPa)	TS (MPa)	EL (%)		
TMCP	20	358	678	63	—	189
	-269	1240	1659	37	194	—
ST	20	298	627	70	—	166
	-269	1145	1558	39	213	—

4. Conclusions

The effect of TMCP on strengthening in the 22Mn-13Cr-5Ni austenitic stainless steel plate for cryogenic use has been systematically investigated by a hot compression test. On the basis of the present results, the steel plate of thickness 70 mm was produced on a large scale under optimum TMCP conditions, the plates were investigated for the mechanical properties.

The results are summarized as follows:

(1) The finish-rolling temperature, cooling rate and start-cooling temperature are the factors controlling the recrystallized austenite grain size. Austenite grain becomes finer as the finish-rolling temperature decreases, the cooling rate and the start-cooling temperature increase in the recrystallization region. In this case, to lower the finish-rolling temperature plays a part in refining the recrystallized austenite grain, and increase in the cooling rate and start-cooling temperature play parts in restraining the recrystallized austenite grain growth. Austenite grain of the 22Mn steel can be refined up to 10 μm by the TMCP and consequently, hardness can be increased about 30 in HV.

(2) The increase in the 0.2 % yield and tensile strengths by applying TMCP is about 50 MPa at room temperature and about 100 MPa at -269°C ,

though the fracture toughness at -269°C is slightly reduced.

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