Effect of Laves Phase on the Properties of Ti and Nb Stabilized Low C, N-19%Cr-2%Mo Stainless Steel Sheets*

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Synopsis

An investigation has been made on the effects of the annealing conditions and cooling rates on the Charpy impact value and the other mechanical properties of Ti and Nb stabilized low C, N-19%Cr-2%Mo stainless steel sheets. The precipitation of Laves phase has a great influence upon the mechanical properties of the steel. Therefore its precipitation behavior has been investigated in connection with the manufacturing process. On the basis of these studies, the best process has been proposed.

I. Introduction

A new type ferritic stainless steel has been developed by lowering C and N contents with the help of recent advancements in steel refining process and by adding suitable amount of Cr, Ti, Nb and Mo in order to improve stress-corrosion resistance and mechanical properties. It is well known, however, that the additions of such carbo-nitride forming elements stimulate the precipitation of intermetallic compounds and deteriorate corrosion resistance in high-purity ferritic stainless steels.¹⁾ The intermetallic compounds have been also known to affect the mechanical properties of high-purity ferritic stainless steels,²⁾ but the detailed analysis on the effect has not yet been made. This report will describe the effect of the Laves phase on the mechanical properties of commercially available Ti- and Nb-stabilized low C, N-19%Cr-2%Mo stainless steel sheets. The effects of heat treatment and cold rolling on the precipitation behavior of the Laves phase will be also discussed.

II. Experimental Procedures

1. Materials, Heat Treatments and Cold Rolling Conditions

Hot rolled sheets of 3.8 mm in thickness were studied. The chemical composition of the steel is given in Table 1.

Annealing of hot-rolled sheets was carried out in the temperature range of 900 to 1 200 °C, and the sheets were cooled at average cooling rates of 1.4×10^2 , 2.0×10^0 or 2.4×10^{-2} °C/sec between 950 °C and 400 °C. After annealing, the sheets were cold rolled by reduction of 0 to 92 %. The cold-rolled sheets were annealed again in the temperature range of 900 to

1 000 °C, and cooled at average cooling rates of 4.2×10^2 , 6.9×10^0 or 2.3×10^{-2} °C/sec between 920 °C to 400 °C.

2. Mechanical Tests

Tensile test was carried out at a strain increasing rate of 40 %/min at 22 ± 2 °C, using JIS No. 13B type specimens on a 10-t capacity Instron universal tensile tester.

The $\bar{r}$ value of the sheet was determined as an average of r values in the L , 45 deg and T directions, namely

$$\bar{r} = (r_L + 2r_{45^\circ} + r_T)/4.$$

Each r value was calculated from the change in width, which was the average of three points to an accuracy of 1/100 mm, and also from the change in length between the 20 mm gauge marks at the center of specimen width after 15 % tension.

Surface roughness was measured on the specimen after 20 % tension by the Kosaka stylus method. The radius of stylus tip was 5 μ m. Ridging was evaluated from the maximum surface roughness in the 20 mm gauge length. Orange peel was measured by the H max method in accordance with JIS B0601.

Charpy impact values were obtained using JIS No. 4 test specimens of 2.5 mm width.

3. Microstructural Analysis

The extraction replica and thin foil techniques were used for electron microscope observations. Extraction replica specimens were etched at 100 mA/cm² in a 3.6 % zinc chloride-10 % hydrochloric acid-methanol solution, and the replicas were prepared by the two-stage Formvar-carbon replica method. Thin foils were prepared by chemical polishing to a thickness of 0.05 mm in a 3:2 solution of phosphoric acid and sulfuric acid, followed by electrolytic polishing at 70 V in a 12:1 solution of acetic acid and perchloric acid. An H500 electron microscope (accelerating voltage: 125 kV) was used to observe microstructures and precipitates, and to identify the precipitates by electron diffraction. A HD-1 000 high voltage electron microscope (accelerating voltage: 1 000 kV) was

Table 1. Chemical composition of low C, N-19%Cr-2%Mo stainless steel sheets. (wt%)

Materials	C	Si	Mn	P	S	Cr	Mo	Ti	Nb	N
LC, N-19%Cr-2%Mo	0.008	0.08	0.14	0.021	0.009	18.64	1.97	0.32	0.30	0.0079

($t=1.0$ mm).

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also employed to verify the presence of fine precipitates.

A JSM-50A scanning electron microscope was used for fractography. The accelerating voltage was 25 kV, and the sample current was $7 \times 10^{-5} \mu\text{A}$.

An EMX-SM electron probe microanalyzer was used to qualitatively analyze the precipitates for elements contained. The accelerating voltage was 20 kV. The sample current and exposure time were 0.01 μA and 40 sec for Fe and Cr; 0.1 μA and 40 sec for Mo, Ti and Nb; 0.1 μA and 160 sec for C and N.

In addition, the precipitates were separated by potentiostatic electrolysis at 0 mV *vs.* SCE in a solution of 10 % acetylacetone, 1 % tetramethyl ammonium chloride and methanol. The amount of precipitates was determined from the resultant extraction residue. The extraction residue was also used to identify the precipitates by X-ray diffraction.

III. Experimental Results and Discussions

1. Effects of Heat Treatments on the Properties of Hot-rolled and Cold-rolled Sheets

Figure 1 shows the effect of annealing temperature

Fig. 1. Effect of annealing temperature of as-hot-rolled sheets on the properties of cold-rolled and annealed sheets. (Cold reduction: 74 %, Annealing condition: 2 min at 900 °C)

of as-hot-rolled sheets on the properties of cold-rolled and annealed sheets. Hot-rolled sheets were heated for 2 min at each temperature between 900 °C and 1 200 °C, cooled in air, cold rolled by 74 %, and reheated for 2 min at 900 °C followed by air cooling. With an annealing temperature above 1 000 °C, the tensile strength increases while the elongation decreases. As annealing temperature increases, the $\bar{r}$ value exhibits the maximum at 950 °C but shows a lower value at 1 000 °C and higher. Planar anisotropy changes from *V* type at 950 °C to inverted-*V* type at 1 000 °C and becomes linear type at 1 200 °C. As annealing temperature rises, ridging is improved but orange peel becomes worse. The surface roughness, represented by the sum of ridging and orange peel, showed the minimum at annealing temperature of 950 °C.

Figure 2 shows the effect of annealing condition after cold rolling on the properties of cold-rolled and annealed sheets. Hot-rolled sheets were heated for 2 min at 950 °C, cooled in air, cold rolled by 74 %, and annealed at 900, 950 and 1 000 °C for various time followed by air cooling. At 1 000 °C, the strength is the lowest, whereas the elongation is the highest. The $\bar{r}$ value is highest at 1 000 °C, but after over 1.5 min

Fig. 2. Effect of annealing condition on the properties of cold-rolled sheets. (Annealing condition of as-hot-rolled sheets: 2 min at 950 °C, Cold reduction: 74 %)

of annealing drops to practically the same level as that at 950 °C. Surface roughness markedly deteriorates at 1 000 °C. Improvement in surface roughness after working is very important for practical purposes. Thus the grain size must be made finer than ASTM No. 6 in order to minimize ridging and to reduce orange peel to a degree acceptable for service. For this purpose, it was decided to heat-treat cold-rolled sheets by annealing at 920 °C for 2 min followed by cooling in air.

The optimum annealing temperature thus determined for this stainless steel is 950 °C for hot-rolled sheets and 920 °C for cold-rolled sheets. High cooling rates are preferred as discussed later.

2. Effect of Cooling Rate on Properties of Hot-rolled Sheets after Heat Treatment

Hot-rolled sheets were annealed at 950 °C for 2 min and cooled at three different rates of 1.4×10^2 , 2.0×10^0 and 2.4×10^{-2} °C/sec to investigate the effect of cooling rate on the impact value of the steel. The results are shown in Fig. 3. As the cooling rate decreases, the transition temperature rises and the Charpy impact value decreases. In relation to the observed toughness, precipitation behavior in this steel was investigated.

Photograph 1 shows the effect of cooling rate on the precipitation after annealing of hot-rolled sheets. At a high cooling rate of 1.4×10^2 °C/sec, no precipitation is recognized at the grain boundaries, but spheroidal precipitates, probably undissolved during annealing, are observed within the grains. At a slow cooling rate of 2.4×10^{-2} °C/sec, large arborescent precipitates are present at the grain boundaries. At an intermediate cooling rate of 2.0×10^0 °C/sec, fine precipitates are recognized at the grain boundaries.

Photograph 2 shows the result of the electron probe microanalysis of the large arborescent precipitate in Photo. 1 (c). It can be seen that this precipitate contains Fe, Cr, Mo, Ti and Nb, but no C. When N coexists with Ti, the N K α line overlaps the elevenfold wavelength of the Ti K α line to make it difficult to verify the presence of N. This analysis suggests the possibility that the arborescent precipitate is not carbide nor nitride but an intermetallic compound containing Fe, Cr, Mo, Ti and Nb.

The electron diffraction analysis of the arborescent precipitate is shown in Photo. 3. From this analysis,

the crystal structure of the precipitate is identified as hexagonal. The lattice parameters a_0 and c_0 are presumed to be 4.78 and 7.84 Å, with the c_0/a_0 ratio being 1.64, which are close to the MgZn₂ (C14)-type Laves phase. Table 2 summarizes observed interspacing values from X-ray diffraction lines of the extraction residues excluding the diffraction lines of TiN and Nb (C, N). The table also includes the values obtained from Photo. 3 and other electron diffraction patterns and calculated values by assuming precipitates having the hexagonal crystal structure mentioned above. These results as well as the qualitative electron probe microanalysis lead to the conclusion that the hexagonal precipitate is the MgZn₂ (C14)-type Laves phase in the form of (Fe, Cr)₂ (Mo, Ti, Nb).

Photograph 4 shows the electron diffraction pattern

Fig. 3. Effect of cooling rate on the transition curve and the percent brittle fracture of the as-hot-rolled sheets. (Annealing condition: 2 min at 950 °C)

(a) Cooling rate: 1.4×10^2 °C/sec (b) Cooling rate: 2.0 °C/sec (c) Cooling rate: 2.4×10^{-2} °C/sec

Photo. 1. Precipitates in sheets annealed at 950 °C.

Photo. 2.
Characteristic X-ray images of precipitates in sheets annealed at 950 °C.
(Cooling rate: 2.4×10^{-2} °C/sec)

Photo. 3. Identification of precipitate in sheet annealed at 950 °C. (Cooling rate: 2.4×10^{-2} °C/sec)

Table 2. X-ray and electron diffraction data.

Observed (Å)			Calculated (Å)	
X-ray diffraction ¹⁾	Intensity	Electron diffraction ²⁾	Laves phase ³⁾	Indices (h·k·l)
—	—	4.14	4.14	(00·1)
—	—	3.65	3.66	(01·1)
2.442	m	2.43	2.39	(11·0)
2.286	S	2.28	2.29	(11·1)
2.195	VS	2.19 2.18	2.21	(01·3)
2.073	W	—	2.07	(02·0)
2.042	W	—	2.04	(11·2)
2.027	m	2.00 1.98	2.00	(02·1)
1.953	W	—	1.96	(00·4)

1) Residue extracted from as-hot-rolled sheet by electrolysis.
2) Precipitates of sheets annealed at 950°C and furnace-cooled.
3) Provisional unit cell: Hexagonal $a_0=4.78$, $c_0=7.84$, $c_0/a_0=1.64$.

of the fine precipitates at the grain boundaries in Photo. 1 (b). Because of their small quantity, the fine

precipitates provide incomplete Debye–Scherrer rings, but the electron diffraction analysis suggests that the precipitates are the Laves phase similar to that obtained in the sheet cooled at a rate of 2.4×10^{-2} °C/sec.

Figure 4 shows the effect of cooling rate of hot-rolled sheets on the Charpy impact value at room temperature and the amount of precipitates. As the cooling rate decreases, the amount of precipitates increases and the Charpy impact value decreases. In order to study the precipitation behavior of the Laves phase more precisely, hot-rolled sheets were heated for 1 hr at temperatures between 500 °C and 1 000 °C followed by quenching into water (which gives an average cooling rate of 1.4×10^2 °C/sec if heated at 950 °C) and then the change in the amount of the resultant precipitate was measured. The results are given in Fig. 5. Although there is little or no precipitation in the as-hot-rolled condition, the quantity of the precipitates reaches the maximum around 700 °C and decreases above 900 °C. Approximately 0.3 % of precipitates is present in the as-hot-rolled condition and at reheating above 900 °C. Such precipitates are probably TiN or Nb (C, N), which hardly dissolves even in the temperature range of 900~1 000 °C. The Laves phase precipitates in a far larger quantity during annealing around 700 °C

Fig. 4. Effect of cooling rate on the Charpy impact value at room temperature and the residue extracted by electrolysis of the as-hot-rolled sheets. (Annealing condition: 2 min at 950 °C)

Fig. 5. Precipitation behavior of Laves phase with isochronal annealing for 1 hr at each temperature.

than during slow cooling from 950 °C. The annealing at 700 °C results in the precipitation of numerous fine particles from the grain boundaries into the grains in the form of a band as shown in Photo. 5. The Charpy impact test reveals that the fine Laves phase,

in spite of its large quantity, does not reduce the impact value so drastically as the large Laves phase that precipitates at the grain boundaries upon slow cooling. The toughness of the hot-rolled sheet heated for 2 min at 950 °C decreases as the cooling rate decreases. This effect can be explained in terms of growth of Laves phase in number and size at grain boundaries. It is reported by Bhandarkar *et al.*³⁾ that the Laves phase precipitated at grain boundaries drastically reduces the toughness at ambient temperature in Fe-Ta-Cr alloys. In order to avoid production problems like strip breakage during cold rolling, the cooling rate of hot-rolled sheets after heat treatment must be maintained above 2.0 °C/sec, and forced air cooling is necessary.

3. Effect of Cooling Rate on Properties of Cold-rolled and Annealed Sheets

Figure 6 shows the effect of cooling rate after annealing on the tensile properties of cold-rolled and annealed sheets. As the cooling rate becomes lower, the strength increases and the elongation decreases. As shown in Fig. 7 the slower cooling rate results in the increase in the amount of precipitates. Therefore the large amount of precipitates deteriorates elongation, as can be seen from Fig. 8 which shows the relationship between the amount of precipitates and elongation. As already mentioned before, the precipitates grow in both size and number, with the decrease in cooling rate. In the case that the cooling rate is at 2.3×10^{-2} °C/sec, large particles of various shapes are observed at grain boundaries and within grains. Scanning electron and optical microscopies reveal that the fracture mode in the tensile test is ductile in every specimen for all the three cooling rates. However, as the cooling rate decreases, the number of small voids increases and irregularities on the fracture surface become smaller. It is also found that voids nucleate at a small amount of large TiN particles when the cooling rate is high and that they develop from the Laves phase in the matrix and at grain boundaries when the cooling rate is low. It is generally known that the ductile fracture of metallic materials is caused by those voids which nucleate at the second-phase particles dispersed in the matrix and eventually grow and coalesce as deformation proceeds. Gladman *et al.*⁴⁾ have suggested that the fracture strain ϵ_f is related

(a) Bright field image (b) Electron diffraction image (c) Electron diffraction data
Photo. 5. Identification of precipitate in sheet annealed at 700 °C for 1 hr. (Cooling rate: $1.4 \times 10^2 \text{ }^\circ\text{C/sec}$)

Fig. 6. Effect of cooling rate on the tensile properties of cold-rolled and annealed sheets. (Cold reduction: 74 %, Annealing condition: 2 min at 920 °C)

Fig. 7. Effect of cooling rate on the amount of the residue extracted by electrolysis in the cold-rolled and annealed sheets. (Cold reduction: 74 %, Annealing condition: 2 min at 920 °C)

Fig. 8. Relationship between the elongation and the residue extracted by electrolysis of cold-rolled and annealed sheets. (Cold reduction: 74 %, Annealing condition: 2 min at 920 °C)

to the sum of the volume fractions of sulfide inclusion, oxide inclusion and carbide as follows:

$$\epsilon_f = 1/(\text{const.} + f_s + f_o + f_c) \dots\dots\dots(1)$$

The relationship between the amount of precipitates and the elongation shown in Fig. 8 exhibits a parabolic curve as predicted from Eq. (1). Therefore it is concluded that the sharp drop in elongation, which is observed after slow cooling of annealed sheet, is due to the precipitation of the Laves phase in a great amount.

A large amount of precipitation of the Laves phase increases the strength. High-voltage electron microscopy reveals that there exist fine precipitates of about 200 Å as well as the Laves phase of about 0.5 μm, as shown in Photo. 6. Those fine precipitates probably act to increase the strength and they are considered to be also the Laves phase but their fineness makes their identification impossible. Strengthening due to such a fine Laves phase is reported by Jones and West⁵⁾ for an Fe-1.8%Nb steel.

The orientation relationship between the rodlike Laves phase and the matrix in cold-rolled and annealed and slowly cooled sheets is derived from Photo. 7 as:

$$(0001)_{\text{Laves}} // (110)_{\alpha}, [11\bar{2}0]_{\text{Laves}} // [\bar{1}11]_{\alpha} \dots\dots\dots(2)$$

In Photo. 7 the direction of the incident electron beam is $[11\bar{2}0]_{\text{Laves}}$, $[\bar{1}11]_{\alpha}$. Even if the electron beam enters not exactly but nearly parallel to this direction, there may appear a diffraction pattern with $[11\bar{2}0]_{\text{Laves}}$ and $[\bar{1}11]_{\alpha}$ as the zone axis. If it is the case the orientation relationship might be rotated approximately 5 deg from the above-mentioned relationship as:

$$(0001)_{\text{Laves}} // (110)_{\alpha}, [1\bar{2}10]_{\text{Laves}} // [001]_{\alpha} \dots\dots\dots(3)$$

Trace analysis strongly suggests that the habit plane, if any, must be $\{110\}_{\alpha}$ and the preferred growth orientation, if any, must be $\langle 001 \rangle_{\alpha}$. Relationship (2) was found by Speich,⁶⁾ and Jones and West⁵⁾ in Fe₂Nb, whereas relationship (3) was suggested by

Photo. 6. Transmission electron micrograph of fine precipitates in cold-rolled and annealed sheet. (Cold reduction: 74 %, Annealing condition: 2 min at 920 °C)

Hornbogen.⁷⁾ The habit plane is reported to be $\{110\}_{\alpha}$ in each case. Cocks and Borland⁸⁾ recently reported a relationship rotated 30 deg around $[\bar{1}11]_{\alpha}$ with respect to relationship (2) as:

$$(11\bar{2}0)_{\text{Fe}_2\text{Nb}} // (\bar{1}11)_{\alpha}, [0001]_{\text{Fe}_2\text{Nb}} // [211]_{\alpha} \dots\dots\dots(4)$$

and they reported that the habit plane is $\{111\}_{\alpha}$. Orientation relationships (2) and (3) are completely different from (4), and the habit planes are also different. As admitted by Cocks and Borland,⁸⁾ it may be reasonable to consider that there can be several orientation relationships rather than only one orientation relationship.

4. Effect of Cold Rolling Reduction on Properties of Cold-rolled and Annealed Sheets

Figure 9 shows the effect of cold rolling reduction on the properties of cold-rolled and annealed sheets. As the cold rolling reduction increases, the strength tends to increase and the elongation does to decrease. There is, however, a unique point near 20 % cold rolling reduction, where the strength extremely increases and exhibits a peak and the elongation decreases and shows a bottom. There is no such point for the $\bar{r}$ value or grain size. To clarify this reason, the following experiments were carried out.

The effects of cold rolling reduction on the elongation and amount of precipitates are shown in Fig. 10. At the 20 % reduction where the elongation sharply deteriorates, the amount of precipitates extremely increases. Optical and electron microstructures of the steel at this reduction are shown in Photo. 8, in which a large amount of precipitates is observed along the grain boundaries and slip bands. It is found from Photo. 8 and Fig. 11 that most of the precipitates are formed prior to recrystallization on heating. Electron diffraction also identifies the precipitates as Laves phase. These results indicate that at the 20 % reduction a very large amount of the Laves phase is precipitated to increase the strength and decrease the elongation of the steel.

The reason why such a large amount of the Laves phase is precipitated at the 20 % cold rolling reduction is not clear. There is a possibility that an autocatalytic reaction occurs to substantially increase the precipitation rate of the Laves phase, as observed by

Photo. 7. Transmission electron micrograph, of cold-rolled and annealed sheet, selected area electron diffraction pattern from Laves phase and its interpretation. (Cold reduction: 74 %, Annealing condition: 2 min at 920 °C, Cooling rate: 2.3×10^{-2} °C/sec)

(a) Optical microstructure (b) Electron microscopic structure (c) Electron diffraction data
Photo. 8. Optical and electron microscopic structures and identification of precipitate in 20 % cold-rolled and annealed sheets. (Annealing condition: 2 min at 920 °C)

Fig. 9. Effect of cold reduction on the properties of cold-rolled and annealed sheets. (Annealing condition: 2 min at 920 °C)

Fig. 10. Effect of cold reduction on the elongation and residue extracted by electrolysis of cold-rolled and annealed sheets. (Annealed condition: 2 min at 920 °C)

Fig. 11. Precipitation of Laves phase. The alloy was quenched at each temperature during heating up. (Cold reduction: 20 %)

Jones *et al.* in case of precipitation of the Laves phase in Fe-Ta alloys.⁹⁾ The rate of the autocatalytic reaction under which the nucleation and growth of particles occur at dislocations will greatly depend on the formation rate of dislocations which act as new precipitation sites. Cold working of approximately 20 % is considered to give a high enough dislocation density to nucleate particles at dislocations, and also gives a low enough dislocation density to facilitate the formation of new dislocations due to the stress field created by precipitates. As the cold rolling reduction is increased, the precipitates along the slip band disappear and the Laves phase particles uniformly precipitate at

grain boundaries and within grains, and the amount of precipitates decreases.

Figure 9 indicates that cold rolling reduction of above 80 % is preferred to obtain a better *r* value.

IV. Summary

The present study has examined the effects of the Laves phase on the properties of low C, N-19%Cr-

2%Mo stainless steel sheets and effects of heat treatment and cold rolling on the precipitation behavior of the Laves phase. The results obtained are the following:

(1) The optimum manufacturing conditions for cold-rolled and annealed sheets of this steel are as follows. Hot-rolled sheets are annealed at 950 °C for 2 min and rapidly cooled, then cold-rolled 80 % or more to improve the r value. Cold rolled sheets should be annealed at 920 °C for 2 min and rapidly cooled.

(2) The Laves phase precipitated in this steel is MgZn_2 (C14) type $(\text{Fe, Cr})_2(\text{Mo, Ti, Nb})$ ($a_0=4.78 \text{ \AA}$, $c_0=7.84 \text{ \AA}$, $c_0/a_0=1.64$). There is a strong possibility that the orientation relationship between the matrix and the Laves phase is $(0001)_{\text{Laves}}//(\bar{1}10)_\alpha$, $[11\bar{2}0]_{\text{Laves}}//[\bar{1}11]_\alpha$. This Laves phase precipitates most rapidly around 700 °C and dissolves above 900 °C.

(3) The Laves phase which dissolves in solid solution at an annealing temperature of over 900 °C precipitates first at grain boundaries and then into grains during cooling. As the cooling rate becomes lower, the amount of Laves phase precipitates increases. This large amount of the Laves phase increases the strength and decreases the elongation. The large Laves phase, which precipitates at grain boundaries when the steel is cooled slowly from 950 °C, decreases the toughness of the steel. These ductility and toughness can be improved by heating above 900 °C to dissolve the Laves phase in solid solution, followed by rapid cooling such as forced air cooling.

(4) A unique phenomenon is recognized that cold rolling of approximately 20 % markedly promotes the precipitation of the Laves phase during annealing. A large amount of the Laves phase precipitated in such

a cold rolling reduction increases the strength but decreases the elongation. Therefore, cold rolling reduction of about 20 % should be avoided for this steel.

The presence of the Laves phase is first confirmed in the low C, N-19%Cr-2%Mo stainless steel to which Ti and Nb are added in combination. It is necessary to study the effect of the Laves phase on the corrosion resistance of the steel in connection with the precipitation behavior of the Laves phase.

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