Optimal Chemical Composition in Fe-Cr-Ni Alloys for Ultra Grain Refining by Reversion from Deformation Induced Martensite

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A thermomechanical treatment, which applies reversion from deformation induced martensite (α') to ultra grain refining of austenite (γ), was proposed for metastable austenitic stainless steels. To determine optimal steels for the treatment, the effect of chemical composition on the γ - α' transformation behavior during cold rolling and the α' - γ reversion behavior by successive annealing was investigated in Fe-Cr-Ni ternary alloys. An ultra fine γ grain structure was obtained when steels satisfied the following three compositional conditions:

(1) Metastable γ should be almost completely transformed to α' during cold rolling at room temperature. The amount of α' induced by 90% cold rolling can be estimated by the Ni equivalent (Ni+0.35Cr). For steels with the Ni equivalent of less than 16.0 mass%, over 90 vol% of γ transforms to α' during 90% cold rolling at room temperature.

(2) Most of deformation induced α' must revert to γ again at relatively low temperatures where grain growth is difficult to occur. When the Cr equivalent (Cr-1.2Ni) is less than 4.0 mass%, most α' induced by the 90% cold rolling reverts to γ through 873 K-0.6 ks annealing. Retained α' is less than 10 vol%.

(3) The *Ms* temperature of the reversed γ obtained through 873 K–0.6 ks annealing should be below room temperature. When the Ni equivalent (Ni+0.65Cr) of steels is more than 19.7 mass%, the reversed γ is stable at room temperature. For the Fe–Cr–Ni ternary alloys which satisfy these three conditions, an ultra fine γ grain structure might be obtained through the reversion from deformation induced α' . For example, γ grains of 0.5 μ m were observed in a 15.5%Cr–10%Ni steel which was subjected to 90% cold rolling and subsequent 873 K–0.6 ks annealing.

KEY WORDS: austenitic stainless steel; nickel equivalent; chromium equivalent; deformation induced martensite; reversion; *Ms* temperature; thermomechanical treatment; grain refining.

1. Introduction

Austenitic stainless steels, despite their excellent corrosion resistance, have low yield strength. Grain refining is an effective means to increase the yield strength. In general, recrystallization after cold or hot rolling is applied to grain refining of austenitic stainless steels because they do not undergo phase transformations on heating or cooling. The strengthening by grain refining, however, is limited because the recrystallization temperature is so high that the smallest austenite (γ) grain size obtainable is several micrometers. For instance, the recrystallization temperature of 18Cr-8Ni stainless steel is above 1 200 K and the minimum grain size obtained through recrystallization treatment is at best about 10-30 μ m (ASTM Nos. 10-8).¹⁾ The high yield strength required in the steel sheets for railroad vehicles application has, to date, been obtained by cold rolling. The strengthening is mainly due to the deformation induced transformation from γ to bcc martensite (α'). SUS 301 and SUS 201 are the typical metastable austenitic steels in which the strength is controlled by cold rolling^{2,3)} and the volume fraction of α' significantly affects the yield strength of cold rolled sheets. However, it is difficult to precisely control the strength because the $\gamma-\alpha'$ transformation is very sensitive not only to deformation rate but also to deformation temperature.⁴⁾

Steels containing α' phase, on the other hand, abruptly soften because of $\alpha'-\gamma$ reversion when they are heated to above 800 K. Much investigation of the reversion mechanism has been done⁵⁻¹³⁾ to clarify this softening behavior. Coleman and West¹²⁾ studied the reversion behavior of α' induced by subzero rolling for a 16%Cr-12%Ni steel and found that the grains of γ reversed from deformation induced α' were smaller than those of γ recrystallized from cold rolled γ .

We have already proposed the thermomechanical treatment¹⁴⁾ to obtain ultra fine γ grains through the $\alpha' - \gamma$ reversion: Metastable γ is almost transformed to full α' by heavy cold rolling and then the α' is reversed to γ by annealing at around 850 K. In this paper, the chemical composition range optimal for the grain refining

treatment was determined for the typical Fe-Cr-Ni austenitic stainless steels.

2. Experimental Procedure

2.1. Materials and Heat Treatment

Electrolytic iron, nickel and pure chromium chips were induction-melted and then cast into a $30 \times 50 \times 100$ mm metallic mold in a vacuum of under 0.1 Pa. Chemical compositions of steels used are listed in **Table 1**. The content of interstitial impurities was small. One and a half kilogram ingots were homogenized at 1523 K for 18 ks and rolled to about 10 mm thick plates (70% reduction) at 1323 K. The steel plates were cold rolled to sheets of different thickness, solution treated in an argon atmosphere at 1373 K for 1.8 ks and then aircooled. Reversion treatment was carried out using a salt bath which enables rapid heating (300 K/s) of specimens. Specimens were isochronally annealed 0.6 ks at various temperatures between 673 and 1073 K.

2.2. Microstructural Identification

The volume fraction of α' was measured by a magnetic method.¹⁵⁾ A few 1 mm thick sheets were superposed to form a $4 \times 4 \times 30$ mm specimen and the saturation magnetization (*Is*) of the specimen was measured in the 550 kA/m² magnetic field. The volume fraction of α' is given by Eq. (1),

 $\alpha'(vol\%) = (Is/Is^*) \times 100$ (1)

where Is^* is the saturation magnetization of the full α'

Steels	Cr	Ni
14Cr-12Ni	13.89	12.03
15Cr-10Ni	15.18	9.97
16Cr-8Ni	16.11	7.66
16Cr-9Ni	16.06	8.75
16Cr-10Ni	15.59	9.82
17Cr–10Ni	17.49	9.94
	15.78	10.04
	13.93	10.92
	15.07	11.95
	15.08	11.02
	16.37	11.04
	18.43	10.08
	12.75	12.01
	15.19	9.09
	14.28	10.10
	11.88	12.87
	13.00	10.79
	11.39	11.35
	12.52	11.59
	13.38	11.51
	15.91	9.50
	15.38	10.36
	15.53	11.29
	17.38	10.88
Others	C<0.005	N < 0.005
	Si < 0.1	Mn < 0.1

Table 1. Chemical composition of steels used. (mass %)

specimen. The value Is^* is also dependent on the chemical composition of the steel and is represented by the Eq. (2) in the Fe–Cr–Ni ternary alloys.¹⁵⁾

$$Vs^* = 2.154 - 0.0304 \times (Cr + 0.5Ni)^*$$
(2)

The volume fraction of reversed γ was calculated from the decrement in *Is* after reversion treatment: the difference of *Is* between cold rolled and annealed states of the same specimen.

The *Ms* temperature of specimens was also measured by the magnetic method. For the measurement above room temperature, change in the magnetization of specimens was continuously monitored in an alternating magnetic field during period of cooling from annealing temperature. And for the measurement below room temperature, change in α' content was directly measured with a ferrite content meter for specimens cooled to various temperatures below room temperature.

2.3. Transmission Electron Micrography

Microstructures were observed with a 1000 kV transmission electron microscope (TEM). Thin foil specimens for TEM observation were prepared by the jet polishing method in a solution of 40% ethanol, 40% methanol and 20% sulfuric acid. For the measurement of γ grain size, an apparent grain size was determined from transmission electron micrographs with reference to the ASTM standards of grain size (\times 100) and then it was converted to the real size taking into account to magnification of the micrographs.

3. Results and Discussion

3.1. Outline of Thermomechanical Treatment for Ultra Grain Refining

To obtain ultra fine γ grains, three conditions must be satisfied for the thermomechanical treatment given in **Fig. 1**:

(1) Metastable γ should be almost completely transformed to α' by cold rolling at room temperature, because the grain size of γ recrystallized from cold rolled γ (γ_c) is not small.¹²)

(2) Reversion temperature of α' to γ must be controlled as low as possible to suppress grain growth of reversed γ (γ_R).

(3) The transformation temperature of γ_R to α' should be below room temperature to obtain a fully austenitic structure. The stainless steels which satisfy these three

Solution treatment

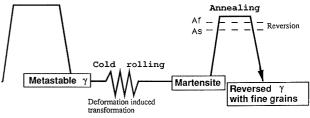


Fig. 1. Thermomechanical treatment to obtain ultra fine structure in metastable austenitic stainless steels.

* The Cr and Ni contents in this paper are expressed by mass%.

conditions will therefore be limited to within a narrow chemical composition range in the Fe-Cr-Ni ternary alloy system.

3.2. Effect of Chromium and Nickel Contents on Deformation Induced Martensitic Transformation

The increasing behavior in the volume fraction of α' during deformation depends on the stability of γ in a steel which Hirayama and Ogirima¹⁶⁾ estimated by the Ni equivalent (Ni + 0.65Cr). Examples are shown in Fig. **2**. The 16Cr–10Ni steel, which has a less stable γ structure than the 14Cr-12Ni steel, easily transforms to α' in the early stage of cold rolling. Perfect transformation, however, is impossible and the volume fraction of α' levels off with a saturation value of over 90 vol% at about 50% deformation. The volume fraction of α' induced by 90% cold rolling is plotted in the Cr-Ni diagram of Fig. 3 for the steels listed in Table 1. Athermal α' is also included in the α' content for steels with the Ms temperature above room temperature. The borderlines of 85, 90 and 95 vol% in α' content are represented as a function of the Ni equivalent (Ni+0.35Cr) in the diagrams. The coefficient of Cr in several Ni equivalents is 0.65^{16} when the γ stability is thermodynamically calculated, 0.45^{17} for Md_{30} temperature and 0.35 for the content of α' induced by 90% cold rolling. These results reveal that the coefficient for Cr decreases with

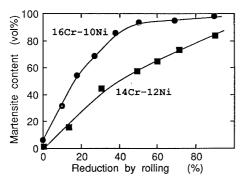


Fig. 2. Relation between reduction by cold rolling and deformation induced martensite content in 16Cr-10Ni and 14Cr-12Ni steels.

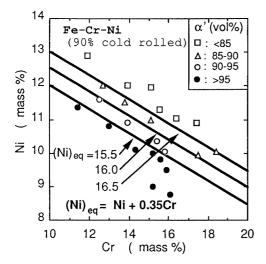


Fig. 3. Effect of Ni and Cr contents on the amount of martensite induced by 90% cold rolling in Cr-Ni stainless steels.

increasing deformation rate, and that the γ stabilizing effect by Cr is smaller in deformation induced transformation than in athermal transformation.

Figure 4 shows the relation between the volume fraction of α' induced by 90% cold rolling and the Ni equivalent (Ni+0.35Cr) in various steels. Data obtained can be plotted along a curve, which is given by Eq. (3) for the Ni equivalent (Ni+0.35Cr) range from 14 to 17 mass%.

$$V_{\alpha'}(\text{vol}\%) = 100 - 2.5 \times (\text{Ni} + 0.35 \text{Cr} - 14)^2 \dots (3)$$

Although the γ stability decreases with decrease in the Ni equivalent, it is difficult to obtain full α' by cold rolling, as shown in Fig. 2. The condition (1) described above, therefore, might be replaced by the following condition: The retained γ_c is reduced to less than 10 vol% by 90% cold rolling. Steels with the Ni equivalent (Ni+0.35Cr) under 16.0 mass% satisfy this condition.

3.3. Effect of Chromium and Nickel Contents on the Microstructure after Reversion Treatment

Chemical composition of steels also affects the reversion from α' to γ . Figure 5 shows the effect of Cr content on the reversion behavior for the 15Cr–10Ni, 16Cr–10Ni and 17Cr–10Ni steels. Ninety percent cold rolled specimens were annealed at several temperatures for 0.6 ks. The amount of α' induced by 90% cold rolling

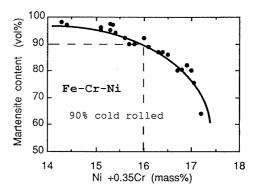


Fig. 4. Relation between Ni equivalent (Ni+0.35Cr) and the amount of martensite induced by 90% cold rolling in Cr-Ni stainless steels.

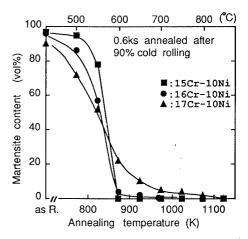


Fig. 5. Decreases in martensite content by martensite to austenite reversion. 15Cr-10Ni, 16Cr-10Ni and 17Cr-10Ni steels cold rolled by 90% reduction were annealed 0.6 ks at selected temperatures.

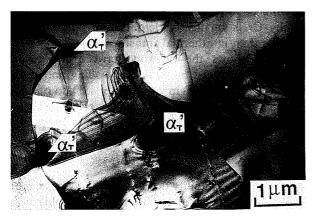


Fig. 6. Transmission electron micrograph of 17Cr-10Ni steel annealed at 923 K for 0.6 ks after 90% cold rolling, showing reversed austenite and ferrite (tempered martensite: α'_T).

decreased as annealing temperature rose because of the $\alpha'-\gamma$ reversion. The steels, however, undergo different reversion depending on Cr content. Reversion of the 15Cr-10Ni steel occurs in a narrow temperature range around 850 K, while that of the 17Cr-10Ni steel cannot be completed unless the steel has been heated to above 1 100 K. With increasing Cr content, reversion finishing temperature rises and the reversion temperature range is broadened. This is mainly attributable to the difference in reversion mechanisms. Increase in Cr content and decrease in Ni content lead to a change in the reversion process from a martensitic to a diffusional mechanism.¹⁸⁾ Figure 6 shows a microstructure of the 17Cr-10Ni steel which has undergone diffusional reversion during 923 K-0.6 ks annealing. A small amount of tempered martensite (α'_T) , which showed a bcc pattern of electron diffraction, can be found in the specimen. During diffusional reversion, atomic movement of Ni and Cr can take place through the boundary between the α' and γ_R phase. Chromium, a ferrite former, should be enriched in α' matrix, while Ni, austenite former, should move towards γ_R , and this will result in formation of Cr rich α'_T particles. Difficulty of perfect reversion in high Cr and low Ni steels is due to the fact that these steels undergo diffusional reversion and α'_T particles, in which the bcc structure is stabilized by Cr, are formed through the reversion process. Most α'_T particles are located at three- and four-grain junctions of γ_R and these might act as obstacles to the grain growth of γ_R .

On the other hand, Fig. 7 shows the effect of Ni content on the reversion behavior in the 16Cr–Ni system steels. Ninety percent cold rolled specimens were also annealed at several temperatures for 0.6 ks. For the 16Cr–8Ni and 16Cr–9Ni steels, the volume fraction of α' decreases with the rise of annealing temperature to 850 K and again increases above 850 K. The increase in the volume fraction of α' is due to transformation from γ_R to fresh α' (α'_F : athermal martensite induced during cooling from the reversion temperature). The increment is larger in the 16Cr–8Ni steel which is less resistant to the athermal transformation than the 16Cr–9Ni steel. Figure 8 shows the $\gamma_R - \alpha'_F$ transformation behavior for 16Cr–8Ni steel. Ninety percent cold rolled specimens were aircooled

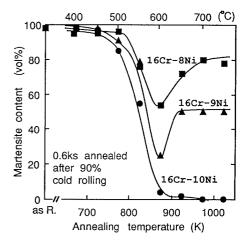


Fig. 7. Changes in martensite content in 16Cr-8Ni, 16Cr-9Ni and 16Cr-10Ni steels as a function of annealing temperature. Specimens were annealed 0.6 ks after 90% cold rolling.

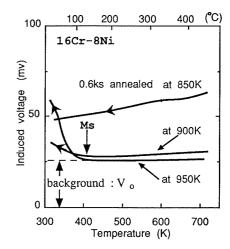


Fig. 8. Changes in magnetization in 16Cr–8Ni steel during air cooling from the annealing temperatures as a function of alternating induction voltage. Induction voltage difference from background corresponds to martensite content in a specimen.

after 0.6 ks annealing at selected temperatures in an alternating magnetic field. The change in the amount of α'_F was detected by measuring magnetization of specimens as alternating induction voltage. Background (V_0) in the alternating induction voltage corresponds to the value for paramagnetic γ single phase specimens and the increment of alternating induction voltage from V_0 corresponds indirectly to the amount of α' , although it is not proportional to the volume fraction of α' . For the specimen annealed at 850 K, high induction voltage was detected at all temperatures below 850 K, because a large amount of α' does not revert to γ but is retained as α'_T after the annealing. The decrease in induction voltage with lowering temperature might be associated with the change in the permeability of α'_T itself. The amount of α'_T decreases with rising annealing temperature and no α'_T was detected in specimens annealed at temperatures above 923 K. For specimens annealed at temperatures over 923 K, however, an abrupt increase in induction voltage was observed under 400 K during cooling. This demonstrates that a part of γ_R again transforms to α'_F

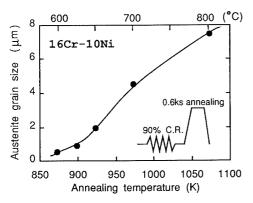


Fig. 9. Relation between annealing temperature and grain size of austenite reversed from deformation induced martensite in 16Cr-10Ni steel.

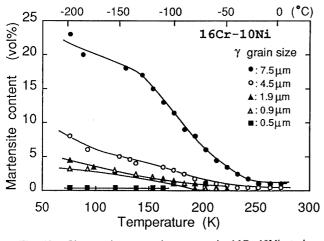


Fig. 10. Changes in martensite content in 16Cr-10Ni steel during cooling from room temperature. Showing grain size dependence of austenite stability at low temperatures.

during cooling and the amount of α'_F increases with rising annealing temperature. Consequently, the constituent phases of the 16Cr-8Ni steel are α'_T and γ_R for specimens annealed below 850 K, α'_F and γ_R for specimens annealed above 950 K and α'_T , α'_F and γ_R for specimens annealed between 850 and 950 K.

In contrast, a fully austenitic structure obtained in the 16Cr-10Ni steel through 0.6 ks annealing above 923 K does not undergo the $\gamma_R - \alpha'_F$ transformation even at room temperature; and the grain sizes of γ_R can be controlled by the annealing temperatures, as shown in Fig. 9. Since the grain sizes increase with rising annealing temperature, ultra fine grains under 1 μ m are obtained through low temperature annealing below 900 K.

Figure 10 shows changes in the content of athermal α' induced during cooling from room temperature to liquid nitrogen temperature in specimens with various γ grain sizes. The amount of α' transformed from γ_R increases with lowering temperature. The increasing behavior, however, tends to be suppressed by refining the γ_R grain size. The specimen with a grain size of 0.5 μ m, which was annealed at 873 K, did not undergo $\gamma_R - \alpha'_F$ transformation even at liquid nitrogen temperature. Furthermore, the *Ms* temperature also depends on the grain size of γ_R . The result is shown in Fig. 11. With refinement of the γ

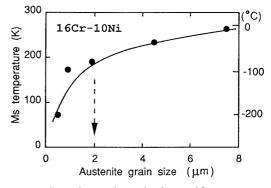


Fig. 11. Effect of austenite grain size on *Ms* temperature in 16Cr-10Ni steel.

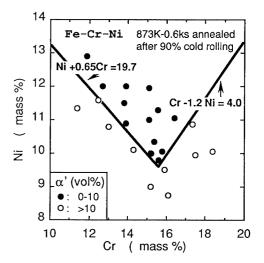


Fig. 12. Effect of Ni and Cr contents on the amount of martensite in specimens annealed at 873 K for 0.6 ks after 90% cold rolling in Cr–Ni stainless steels.

grain size, the *Ms* temperature is lowered. This tendency is significant in specimens with a grain size of under $2 \mu m$. Grain refinement results in γ stabilization, 19^{-21} leading to a lowering of the *Ms* temperature and suppression of athermal α' transformation below this temperature.

For various Cr-Ni stainless steels annealed at 873 K for 0.6 ks after 90% cold rolling, the volume fractions of α' in the specimens are summarized in the Cr-Ni composition diagram as shown in Fig. 12. Steels represented by a solid mark have almost fully austenitic structure (α' content is less than 10 vol%) and the γ_R is stable at room temperature. These steels satisfy the following conditions for chemical composition: The Cr equivalent (Cr - 1.2Ni) is less than 4.0 mass% and the Ni equivalent (Ni+0.65Cr) is more than 19.7 mass%. The former condition is necessary to achieve perfect reversion from α' through 873 K–0.6 ks annealing and the latter is required to obtain a stable austenitic structure at room temperature. For steels with the Cr equivalent of more than 4.0 mass%, many α'_T particles are retained even after the annealing. This may be convenient to suppress the grain growth of γ_R . On the other hand, for steels with the Ni equivalent of less than 19.7 mass%, $\gamma_R - \alpha'_F$ transformation occurs during cooling from the annealing temperature as already shown in Fig. 7. This means that the Ms temperature of steels with the Ni equivalent of 19.7 mass% is around room temperature.

3.4. Optimal Chemical Composition Range for Thermomechanical Treatment to Obtain Ultra Fine Austenite Grains

Figure 13 summarizes the relation between microstructure and Cr–Ni chemical composition for specimens with the reversion treatment of 873 K-0.6 ks after 90%cold rolling. A triangular composition range, where the almost fully austenitic structure is obtained, is determined by the following three conditions:

(1) More than 90 vol% of γ is transformed to α' during 90% cold rolling. (Ni+0.35Cr ≤ 16.0 mass%)

(2) The amount of α' , which does not revert to γ after the reversion treatment of 873 K-0.6 ks, is less than 10 vol%. (Cr - 1.2Ni \leq 4.0 mass%)

(3) The *Ms* temperature of reversed γ is below room temperature. (Ni+0.65Cr \geq 19.7 mass%)

The triangular composition range is narrow in higher Ni and lower Cr composition side, while it is wide on the other side. This means that deformation induced martensitic transformation occurs easily in steels which have higher Cr and lower Ni composition when γ has the same stability against athermal α' transformation.

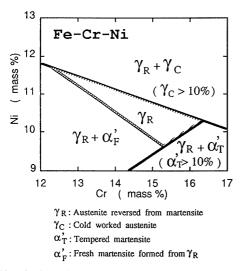


Fig. 13. Optimal chemical composition for ultra grain refining treatment in Cr–Ni steels.

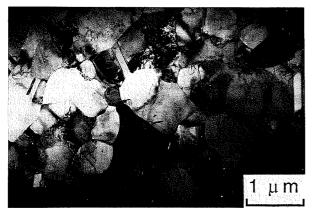


Fig. 14. Transmission electron micrograph of 16Cr-10Ni steel annealed at 873 K for 0.6 ks after 90% cold rolling, showing ultra fine austenite grains with a mean size of $0.5 \,\mu$ m.

For athermal transformation, Hirayama and Ogirima¹⁶⁾ found that the amount of α' abruptly increases when the Ni equivalent (Ni+0.65Cr) is less than 20.7 mass%; it was less than 19.7 mass% in this investigation. This likely results from the difference in γ grain size, and the effect of grain refining on γ stabilization¹⁹⁻²¹⁾ is almost the same as 1% Ni addition in the chemical composition. It plays an important role for enlarging the triangular composition range in Fig. 13.

Figure 14 represents a typical microstructure of ultra fine γ grains obtained for the 16Cr-10Ni steel, which is located in the center of the triangular composition range. The mean grain size of refined is 0.5 μ m and the 0.2% proof stress is increased to 0.7 GPa²² through the ultra grain refining, while it is 0.15 GPa for a solution treated specimen with a mean grain size of 50 μ m.

4. Conclusions

Deformation induced transformation behavior from metastable austenite to martensite and reversion behavior of the martensite were investigated in Fe–Cr–Ni ternary alloys. The results obtained are as follows:

(1) The volume fraction of martensite induced by 90% cold rolling depends on the Ni equivalent (Ni+0.35Cr). Steels with the Ni equivalent of less than 16.0 mass% are almost completely transformed to martensite during 90% cold rolling at room temperature and the amount of retained austenite is less than 10 vol%.

(2) Martensite induced by the 90% cold rolling is almost completely reversed to austenite through 873 K-0.6 ks annealing when steels have a chemical composition of less than 4.0 mass% in the Cr equivalent (Cr-1.2Ni). The amount of martensite, which does not revert to austenite, is less than 10 vol%.

(3) The *Ms* temperature of reversed austenite formed through 873 K–0.6 ks annealing is below room temperature for steels with a chemical composition of more than 19.7 mass% in the Ni equivalent (Ni+0.65Cr).

(4) An almost fully austenitic structure with ultra fine grains is obtained through the reversion from deformation induced martensite when steels satisfy the above three conditions (1) to (3). For instance, an ultra fine microstructure of $0.5 \,\mu$ m austenite grain was obtained in a $15.5 \,\%$ Cr-10 %Ni steel located in the center of the chemical compositional triangle surrounded by three boundary lines as determined on the basis of the above three conditions through the treatment: 90 % cold rolling followed by 873 K-0.6 ks annealing.

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