

DEVELOPMENT OF A SULFIDATION-CORROSION RESISTANT NICKEL-BASE SUPERALLOY FOR FCC POWER RECOVERY TURBINE ROTORS

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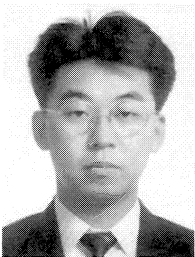
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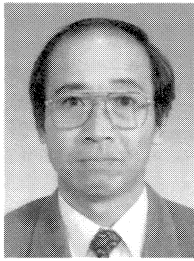
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ABSTRACT

A nickel-base superalloy for FCC power recovery turbine rotors was developed, which shows excellent sulfidation-corrosion resistance, high temperature strengths, and hot workability.

To investigate the influences of the alloying elements of AISI685 on the sulfidation behavior, nickel-base alloys containing each of chromium, cobalt, molybdenum, titanium, and aluminum (the major components of AISI685), and alloys containing multiple such elements were produced and corroded in sulfidizing gas atmospheres at 600°C. It was found that the sulfidation behavior, especially the penetrating behavior into the alloys, was greatly influenced by aluminum, and that the resistance could be improved by increasing the aluminum content in the alloys.

Six types of alloys, in which aluminum had been increased to improve the sulfidation resistance of AISI685 and titanium decreased by the same proportion to maintain the high temperature strengths of AISI685, were forged to test their sulfidation behavior and high temperature strengths. As a result, the alloy with 1.5 percent titanium and 3.0 percent aluminum showed only half the sulfidation depth of AISI685, for the tensile characteristics at 538°C, and at 850°C through 1100°C, equivalent or better than those of AISI685. This means that the alloy with 1.5 percent titanium and 3.0 percent aluminum has twice more sulfidation-corrosion resistance than AISI685, in addition to equal or higher high temperature strengths and hot workability.

A simulation expander rotor disk of 500 mm in diameter was manufactured with the newly developed alloy. The disk was successfully produced without shear cracking or buckling problems. Furthermore, test specimens cut from the disk showed a rupture strength equal to that of AISI685 at 650°C to 750°C.

INTRODUCTION

Gas expander turbines are incorporated in fluid catalytic cracking units (FCC) of oil refineries as power recovery devices. As such, expander turbines are operated in exhausts at 500°C to 700°C, containing corrosive gases such as hydrogen sulfide (H₂S), sulfur dioxide (SO₂), oxygen (O₂), and so on. AISI685 is used for the rotors, a nickel (Ni)-base gamma-prime (γ')-precipitation hardening alloy proven to be very strong and resistant to high temperature corrosion, as a material suitable for machine parts operated at high temperatures. However, several instances of inservice cracking have recently been experienced in rotating blades of expander turbines operated at about 530°C. Therefore, the cause was investigated in order to determine countermeasures (Dowson, et al., 1995). The authors examined the component in question and found that sulfide had been generated along the alloy grain boundary to a considerable depth around the blade root section, leading to the assumption that the cracks had propagated from the sulfidation-corrosion tip by corrosion fatigue, or some corrosion assisted degradation, and caused fracture.

Sulfidation-corrosion with industrial gas turbines has been generally reported as hot corrosion (Stringer, 1979; Birks and

Meier, 1983; Pettit and Giggins, 1987), which is a severe corrosion resulting from by-products related to molten sulfate such as sodium sulfate (Na₂SO₄). However, substances that cause hot corrosion, such as sulfate, sodium, and/or chloride, were not detected in the corroded expander rotor. Therefore, we assumed that the sulfidation-corrosion had been caused by direct reaction between the sulfidizing gases and the alloy. It is reported that Ni-base alloys are subject to catastrophic corrosion through the reaction of a sulfidizing gas and the metal at or over about 700°C (Smeltzer, 1979), even without molten salts such as Na₂SO₄. This is due to the fact that the eutectic point of the Ni and Ni-sulfide (Ni₃S₂) is 635°C (Kullerud and Yund, 1962), thus a melted material is produced at or over that point. However, Ni-base superalloys, in general, are considered to have excellent corrosion resistance in sulfidizing gas atmospheres that are below the eutectic point. Therefore, few cases have been reported in which severe sulfidation-corrosion of alloy grain boundaries had damaged the apparatus.

Since many aspects of the mechanism and behavior of sulfidation-corrosion on the rotating blade of the expander turbine remain unexplained, the authors have been working to:

- Explain the mechanism of corrosion and cracking,
- Develop technology for life prediction of actual machines,
- Develop surface modification technology for improving a sulfidation-corrosion resistance, and
- Develop a sulfidation-corrosion resistant alloy.

Some portions of the studies on corrosion behavior of AISI685 (Yakuwa, et al., 1996) and on the development of the surface modification technology for improving sulfidation-corrosion resistance (Nakahama, et al., 1995) have already been reported. This paper describes the effects of alloying elements on sulfidation behavior of AISI685 and the development of a high temperature sulfidation-corrosion resistant Ni-base superalloy.

EFFECTS OF ALLOYING ELEMENTS ON CORROSION BEHAVIOR

Description of Experiment

AISI685 is a Ni-base superalloy composed of nickel (Ni), chromium (Cr), cobalt (Co), molybdenum (Mo), titanium (Ti), and aluminum (Al) as major alloying elements, as shown in Table 1. To clearly illustrate the effects of the alloying elements on corrosion behavior, various alloys with different components were produced by the argon arc melting method. That is, starting from pure Ni, another element was added to the previous mixture, in accordance with the chemical composition of AISI685, e.g., Ni-20Cr, Ni-20Cr-13.5Co, and so on. Alloys containing zero percent up to 4.5 percent Ti and Al, respectively, were also prepared, to study the effects in detail of variations in the contents. The ingots of the alloys were heat-treated at 1024°C for 24 hours and cooled in the furnace for homogenization. Then, each ingot was sliced into approximately 1 mm thick strips, both surfaces of which were polished to 1 μm diameter diamond paste level and degreased with acetone. Corrosion tests were conducted at 600°C for nine hours through 144 hours. Though the partial pressures of sulfur (pS₂) and oxygen (pO₂) in a 600°C atmosphere of actual machines are 10⁻¹² to 10^{-10.5} atm and 10^{-23.5} atm, respectively, the test gas was adjusted by hydrogen (H₂) and hydrogen sulfide (H₂S) mixtures, so that the pS₂ became either 10⁻¹² or 10^{-10.5} atm and the pO₂ became lower than that of actual machines, in order to give sulfidation priority over oxidation. The corrosion behavior was evaluated by examining the weight gain after the test, observing corrosion morphologies by using a scanning electron microscope (SEM), and identifying corrosion products by x-ray diffract meter (XRD) and electron probe micro analyzer (EPMA).

Table 1. Chemical Composition of AISI685 (Wt. %).

| Ni | Cr | Co | Mo | Ti | Al | C | Zr | B | Fe | Si | S | P | Mn |
|------|------|------|-----|------|-----|------|------|-------|-----|------|-------|-------|-----|
| bal. | 18.0 | 12.0 | 3.5 | 2.75 | 1.2 | 0.02 | 0.02 | 0.003 | 2.0 | 0.15 | 0.015 | 0.015 | 0.1 |
| | 21.0 | 15.0 | 5.0 | 3.25 | 1.6 | 0.10 | 0.08 | 0.010 | | | | | |

Experimental Results

Figure 1 shows the weight gain of the individual alloys corroded at 600°C for 49 hours under $pS_2 = 10^{-10.5}$ atm. The corrosion amount of Ni was dramatically decreased by the addition of 20 percent Cr. As the weight gain of Ni-20Cr-13.5Co alloy is almost equal to that of Ni-20Cr, Co does not have much influence on the corrosion amount of Ni-20Cr. When four percent Mo was added to Ni-20Cr-13.5Co alloy, the weight gain decreased to approximately two-thirds, indicating that the addition of four percent Mo effectively reduced the corrosion. Then, the effects of adding Ti and Al were studied. Three percent Ti was added to Ni-20Cr-13.5Co-4Mo alloy, revealing that the weight gain was almost the same with, or more than, that of Ni-20Cr-13.5Co-4Mo alloy, while the weight gain was almost two-thirds for the alloy containing 1.5 percent Al. Therefore, the addition of three percent Ti to Ni-20Cr-13.5Co-4Mo alloy has almost no effect on the improvement of sulfidation-corrosion resistance (weight gain) or no effect, but that the addition of 1.5 percent Al works. Following this experiment, alloys containing different amounts of Ti and Al were tested to examine in detail the effects of Ti and Al contents in the Ni-20Cr-13.5Co-4Mo alloy. An alloy of 3Ti-1.5Al has a simulated composition as that of AISI685. The weight gain of the 4.5Ti-1.5Al alloy, which contains more Ti than AISI685, was more than that of the 3Ti-1.5Al. On the other hand, 2.5Ti-2.5Al alloy, with higher Al content and lower Ti content, showed approximately half the corrosion of 3Ti-1.5Al.

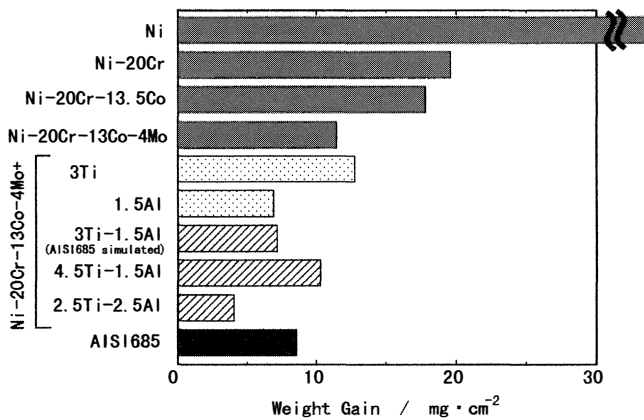


Figure 1. Weight Gain of Ni-Base Alloys Sulfidized in an H_2-H_2S Mixture. (Sulfur partial pressure is $10^{-10.5}$ atm, at 600°C for 49 hours.)

Furthermore, Ni-20Cr-13Co-4Mo alloys having different Ti and Al contents were tested for corrosion behavior at 600°C under $pS_2 = 10^{-10.5}$ and 10^{-12} atm, respectively, to clarify the penetration behavior into the alloys interior. Figure 2 shows the relationship between the depth of penetration into these alloys (including grain boundary penetration) and the contents of Ti-Al.

The penetrated depth here was determined vertically, from the original surface of the alloy through the inner corrosion scale to the tip end of the internal and/or grain boundary penetration. In either atmosphere, a similar trend was observed. That is, comparing the penetrated depths of the alloys containing the same amount of Al, no notable tendency due to Ti content was found. But, comparing the penetrated depths of the alloys containing the same amount of Ti, a tendency was found that the higher the Al content, the less

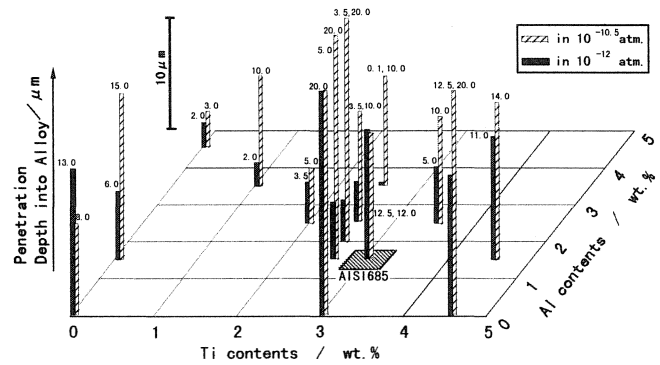


Figure 2. Effect of Ti and Al Contents on Penetration Depth into Ni-20Cr-13Co-4Mo Alloys Sulfidized in H_2-H_2S Mixtures. (Sulfur partial pressure is $10^{-10.5}$ atm, at 600°C for 49 hours.)

deep the penetration became among the alloys examined, except for the alloys that had no Al under $pS_2 = 10^{-10.5}$ atm. Thus, it was found that, by increasing the content of Al, not only the weight gain due to corrosion, but also penetration into the alloy interior would be controlled.

It has been reported that the addition of Ti to Ni-base alloys improves the sulfidation-corrosion resistance in a sulfidizing atmosphere with low oxygen partial pressure at 760°C to 982°C (Bradshaw and Stoltz, 1980; Lai, 1985). On the other hand, it has been reported that the addition of Al to Fe-Cr alloys (Mrowec and Wedrychowska, 1979; Smeltzer, et al., 1982) and Co-Cr alloy (Beigun and Bruckman, 1980) decreases the sulfidation-corrosion rate at or over 700°C, and that adding three percent or four percent Al to Ni-30Cr alloy (Bradshaw and Stoltz, 1980) is also effective to the sulfidation-corrosion resistance at 982°C. Whereas, it has been reported that the effects of adding Al to Ni-25Cr alloy (Beigun and Bruckman, 1980) are so complicated that it simply does not serve to improve the sulfidation resistance. Taking these reports and the results of this experiment into consideration, it is assumed that the effects of adding Ti and Al on sulfidation resistance vary, depending on the alloy composition, environment, temperature, and other conditions. It is also possible to consider that the addition of Ti to Ni-20Cr-13Co-4Mo alloy, in the sulfidizing atmosphere at 600°C, works rather adversely in terms of the weight gain, and shows no clear effect on the penetration into the alloy interior. By contrast, the addition of Al works effectively on both weight gain and the penetration into the alloy interior.

DEVELOPMENT OF A SULFIDATION-CORROSION RESISTANT Ni-BASE SUPERALLOY

Determining the Alloy Composition

Method

The findings in the previous section showed that it is possible to produce an alloy with better sulfidation-corrosion resistance than AISI685 by increasing the Al content, among the alloying elements, by a few percentage points. However, if the material is to be used for practical applications, high temperature strength, ductility, and hot workability, in addition to corrosion resistance, are required.

Since AISI685 is a $\gamma'(Ni_3(Al,Ti))$ -precipitation hardening alloy, the strength characteristics (high temperature strength, ductility, and hot workability) are, for the most part, determined by the quantity of γ' . If the γ' quantity is low, the high temperature strength becomes inadequate, while too much γ' affects the hot workability and makes the forging process difficult. Thus, it is considered possible to maintain the same strength characteristics as those of AISI685 by increasing the Al content to improve the sulfidation-corrosion resistance on the one hand, and by controlling the Ti amount proportionally to the Al increase to keep the γ' content equivalent to that of AISI685 on the other.

Therefore, with each of the components being the same as AISI685, and Al content increased and Ti content decreased, six types of alloys were produced and their sulfidation behavior and strength characteristics were examined. Table 2 lists their chemical compositions. The six types of alloys were melted in an argon atmosphere in an induction furnace and forged with four successive deformations into 20 mm thick plates, the reduction in the thickness is 50 percent, 30 percent, 44 percent, and 56 percent, respectively. After forging operations, heat treatments were applied as follows: solution annealing at 1038°C for four hours followed by oil cooling, stabilizing at 843°C for four hours followed by air cooling, primary aging at 760°C for 16 hours followed by air cooling, and secondary aging at 650°C for 16 hours followed by air cooling. With practical use in mind, we conducted sulfidation-corrosion tests in a close atmosphere to actual machines. Thus, by mixing in nitrogen (N_2) - hydrogen (H_2) - sulfur dioxide (SO_2), the test pO_2 was set at $10^{-23.5}$ atm, approximately equal to that for actual machines, and for the acceleration the sulfur attack, the pS_2 was set at $10^{-5.5}$ atm, which is about five decades higher than that of actual machines.

Table 2. Chemical Composition of Trial Sulfidation-Corrosion Resistance Ni-Base Superalloys.

| | Ni | Cr | Co | Mo | Ti | Al | C | Zr | B | Fe | Si | S | P | Mn |
|-------------|------|-------|-------|------|------|------|-------|------|--------|------|------|-------|-------|------|
| AISI685 | bal. | 19.74 | 13.39 | 4.23 | 3.00 | 1.50 | 0.049 | 0.06 | 0.0045 | 0.94 | 0.06 | 0.001 | 0.002 | 0.01 |
| 2.0Ti-3.0Al | bal. | 19.74 | 13.37 | 4.21 | 2.03 | 2.97 | 0.043 | 0.06 | 0.0043 | 0.99 | 0.03 | 0.001 | 0.002 | 0.01 |
| 2.0Ti-2.5Al | bal. | 19.60 | 13.41 | 4.25 | 2.02 | 2.49 | 0.042 | 0.06 | 0.0045 | 1.00 | 0.04 | 0.001 | 0.001 | 0.01 |
| 2.0Ti-2.0Al | bal. | 19.58 | 13.33 | 4.23 | 2.03 | 2.04 | 0.040 | 0.06 | 0.0043 | 1.01 | 0.05 | 0.001 | 0.001 | 0.01 |
| 1.5Ti-3.5Al | bal. | 19.60 | 13.57 | 4.15 | 1.53 | 3.50 | 0.041 | 0.06 | 0.0042 | 1.00 | 0.04 | 0.001 | 0.001 | 0.01 |
| 1.5Ti-3.0Al | bal. | 19.62 | 13.50 | 4.25 | 1.53 | 3.02 | 0.041 | 0.06 | 0.0045 | 1.02 | 0.04 | 0.001 | 0.002 | 0.01 |
| 1.5Ti-2.5Al | bal. | 19.70 | 13.54 | 4.25 | 1.53 | 2.51 | 0.041 | 0.06 | 0.0046 | 1.01 | 0.05 | 0.001 | 0.002 | 0.01 |

Strength characteristics, including tensile strength, yield strength at 0.2 percent offset, elongation, and reduction in area were determined at 538°C, to compare the values with those characteristics of AISI685 for evaluation. Also, tensile strength, elongation, and reduction in area were determined at 850°C to 1100°C to evaluate the workability during forging.

Test Results

Figure 3 shows the depths of penetration into the alloy, including grain boundary penetration, of the tested alloys and AISI685 corroded for 68 hours in a sulfidizing atmosphere at 600°C.

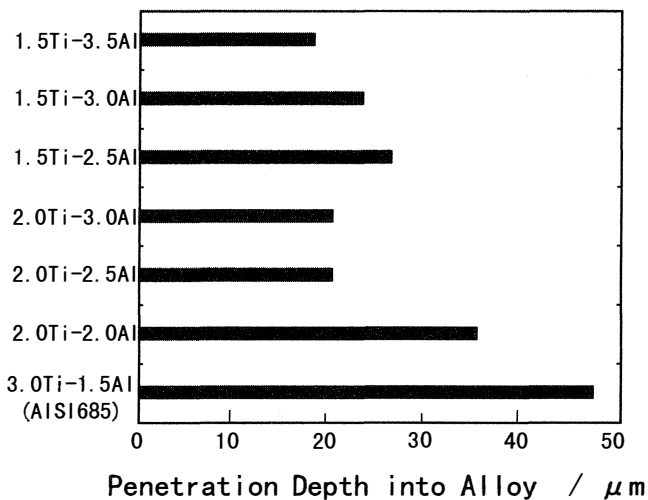


Figure 3. Effect of Ti and Al Contents on the Corrosion Depth into Wrought Ni-20Cr-13Co-4Mo Alloys Exposed to an N_2 -3% H_2 -0.75% SO_2 Mixture. (Sulfur and oxygen partial pressures are $10^{-5.5}$ and $10^{-23.5}$ atm, respectively, at 600°C for 68 hours.)

All of the tested alloys showed less deep penetration than that of AISI685, reflecting their better sulfidation-corrosion resistance. Figure 3 also indicates that penetration into the alloy interior containing more Al was less deep than that for the alloys having the same Ti content. We can thus see that, also under the condition where the oxygen partial pressure is equivalent to the actual operating condition ($pO_2 = 10^{-23.5}$ atm), penetration of sulfidation can be decreased by increasing the Al content by 2.5 percent up to 3.5 percent.

Figure 4 shows the tensile strength, yield strength, elongation, and reduction in area of the test alloys and AISI685 at 538°C. The tested alloys show the same values of tensile strength and yield strength as AISI685. In terms of elongation, only 2.0Ti-3.0Al, the calculated γ' content of which is 34 percent (which is eight percent higher than that of AISI685), showed a lower value than AISI685, while other ones showed the same or higher value than that of AISI685. As for the reduction in area, just like the elongation case, 1.5Ti-3.5Al, the calculated γ' content that is at a high level of 35 percent, showed a value less than that of AISI685, with the other alloys indicating an equal or higher value. Judging from these values, we can say that, in terms of the strength characteristics at 538°C, the increase in calculated γ' content has a greater influence on elongation and reduction in area than on ultimate tensile strength and yield strength at 0.2 percent offset.

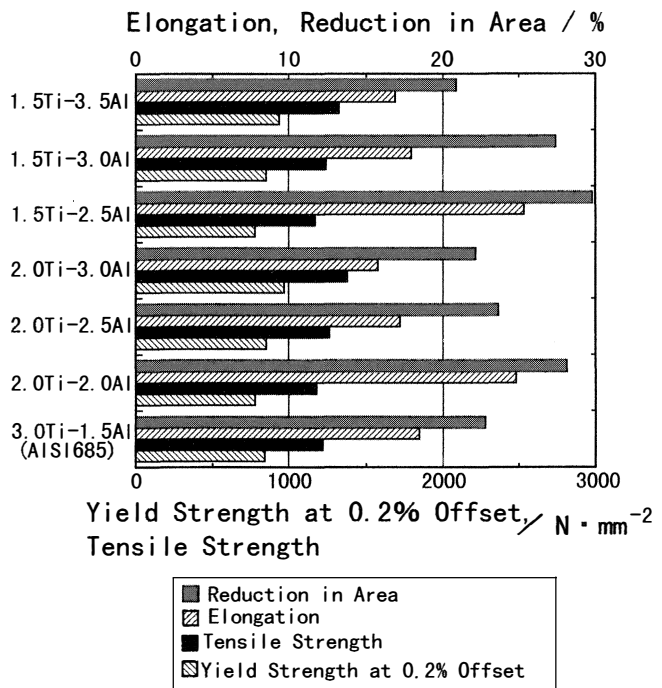


Figure 4. High Temperature Mechanical Properties of Wrought Ni-20Cr-13Co-4Mo Superalloys at 538°C.

Next, we examined the high temperature strength characteristics (tensile strength, elongation, and reduction in area) at 850°C to 1100°C, to check the hot workability. The tensile strength, and the elongation and reduction in area of each alloy are illustrated in Figures 5 and 6, respectively. In terms of elongation, 2.0Ti-2.5Al, 2.0Ti-2.0Al, and 1.5Ti-2.5Al, all of which have a lower calculated γ' content than that of AISI685, showed a slightly lower value than AISI685, while the other ones, 1.5Ti-3.0Al, 1.5Ti-3.5Al, and 2.0Ti-3.0Al, showed the same or higher value than that of AISI685. For elongation at or below 1,000°C, only 1.5Ti-3.5Al indicated a lower value than that of AISI685, but a higher value than that of AISI685 was measured for the other alloys. Furthermore, 1.5Ti-3.5Al and 2.0Ti-2.5Al recorded a reduction in area of less than that of AISI685 at 850°C. From the above, we can say that the alloys,

2.0Ti-2.0Al and 1.5Ti-2.5Al, both of which showed a lower tensile strength, 1.5Ti-3.5Al with a lower elongation value at or below 1,000°C, and 2.0Ti-2.5Al with a lower strength and less reduction in area at 850°C, are slightly inferior to AISI685 in hot workability, but that 1.5Ti-3.0Al and 2.0Ti-3.0Al alloys have equal or superior hot workability to AISI685.

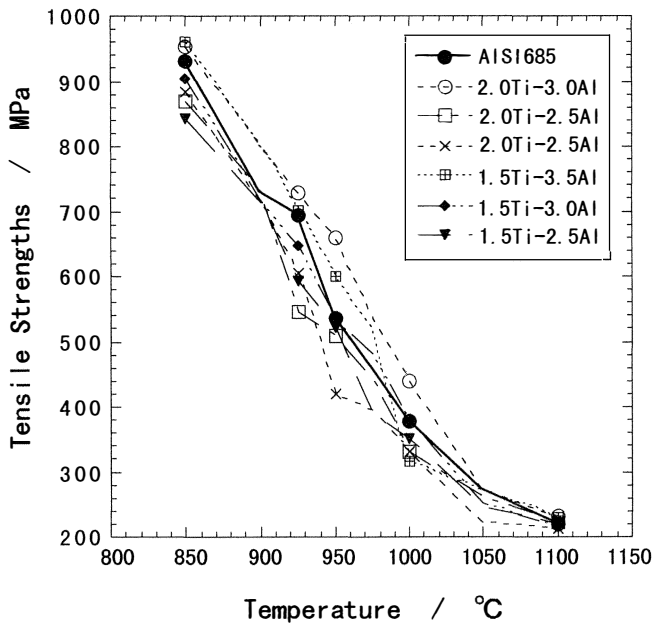


Figure 5. Tensile Strength of Wrought Ni-20Cr-13Co-4Mo Superalloys at a Temperature Range of 850°C to 1100°C.

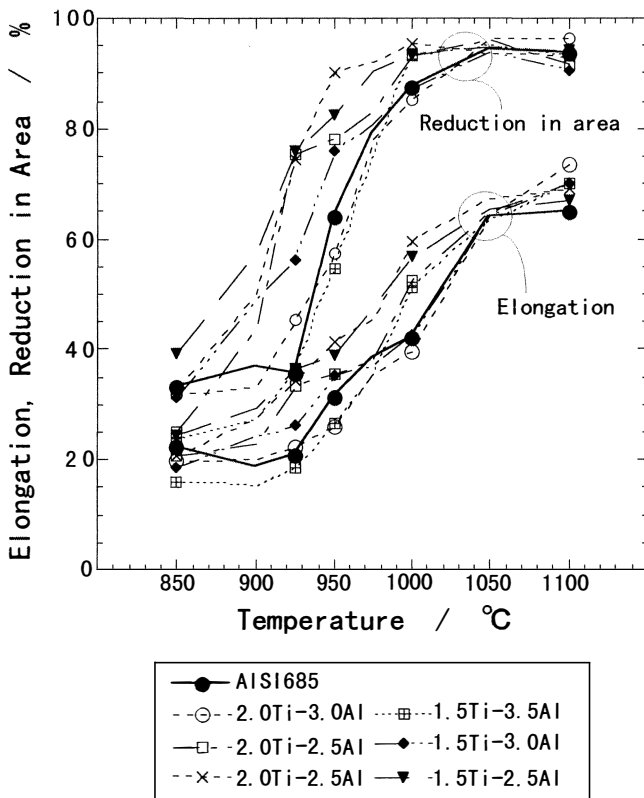


Figure 6. Elongation and Reduction in Area of Wrought Ni-20Cr-13Co-4Mo Superalloys at a Temperature Range of 850°C to 1100°C.

From the results of the sulfidation-corrosion tests, 1.5Ti-3.5Al with the highest Al content may be the best chemical composition for the sulfidation-corrosion resistant Ni-base superalloy for expander rotors. However, in terms of the high temperature strength in the service temperature range and hot workability, 1.5Ti-3.0Al can be judged overall as the best-balanced composition. Therefore, the alloy having this chemical composition was selected as the practical material for the sulfidation-corrosion resistant Ni-base superalloy. In this way, the authors have developed an alloy, which is equal or superior to AISI685 in strength and workability, and twice as sulfidation-corrosion resistant as AISI685.

Manufacturing an Expander Simulation Disk

To examine the possibility of applying the developed alloy to the expander rotors, a simulation disk of 500 mm in diameter was free-forged by upsetting, the same as applied to actual machines. Though the diameter was almost half that of the actual device, the disk was successfully forged without shear cracking, buckling problems, and so on. Test specimens were then cut from the disk in the circumferential direction, to conduct a creep rupture test in air at 650°C to 750°C, according to ASTM standards (Eds. Priemon, et al., 1984). Figure 7 shows the Larson-Miller master curves of the newly developed alloy drawn from the test results and of AISI685 cited from the literature (Eds., Sims, et al., 1987) and data catalogs (Mitsubishi Materials Corporation, 1992; Hitachi Metals Ltd., 1993). This confirms that the new alloy can maintain the creep rupture strength equal to that of AISI685, when used for manufacturing simulation disks for actual expanders.

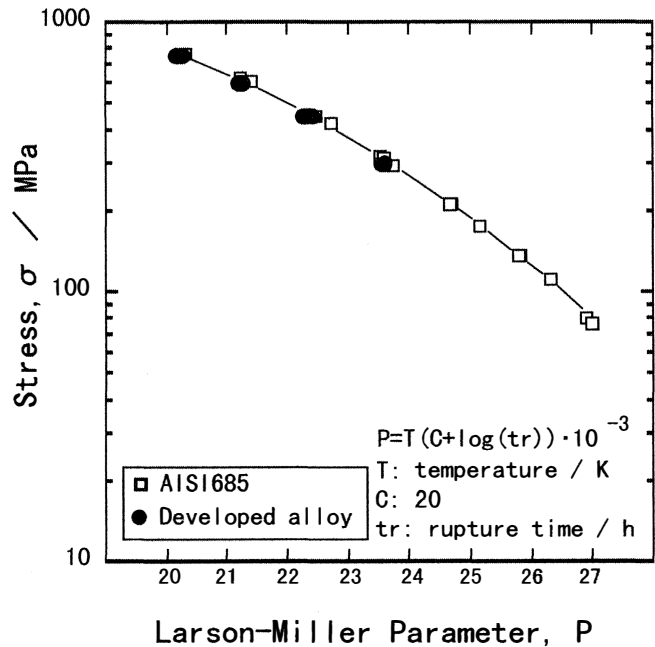


Figure 7. Larson-Miller Master Curves of Developed Sulfidation-Corrosion Resistant Ni-Base Superalloy and AISI685.

Currently, high temperature sulfidation-corrosion tests under stresses, stress relaxation tests, and constant displacement rate tests are being carried out to examine the alloy in more detail.

CONCLUSIONS

- The effects of alloying elements of AISI685 on sulfidation behavior were investigated in H₂-H₂S mixtures at 600°C. An increase in Al content greatly controls the depth of penetration into the alloy interior, including grain boundary penetration due to sulfidation.

- Six types of alloys with a higher Al content and lower Ti content than the contents of AISI685 were tested for sulfidation resistance in an N₂-H₂-SO₂ mixture, high temperature strength, and hot workability. 1.5Ti-3.0Al alloy is excellent overall and well-balanced in terms of sulfidation resistance, high temperature strength, and hot workability, and is judged as the most suitable for the sulfidation-corrosion resistant Ni-base superalloy for gas expander rotors.
- This newly developed alloy has twice the sulfidation-corrosion resistance of AISI685, in addition to excellent strength and hot workability equivalent to those of AISI685.
- A simulation disk of 500 mm in diameter for an actual expander was successfully forged of this new superalloy. Test specimens cut out of the disk were shown to have equivalent rupture strength to AISI685.

Authors are now investigating the characteristics of this material in greater detail by examining the high temperature corrosion behavior under static and dynamic stresses and fracture toughness, in order to apply the material to high speed rotating machines such as gas expanders.

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ACKNOWLEDGEMENT

The authors would like to express their sincere appreciation to Takehiro Ohno of the Metallurgy Laboratory of Hitachi Metals Ltd., who generously supported their approach, especially in creating the test alloys and in evaluating their strength, and to all the staff members of the Yasugi Factory of Hitachi Metals Ltd., for their cooperation.