

γ' Precipitation-hardened Cobalt-free Heat-resistant Steel Strip for Exhaust Gaskets Work at 900°C

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ABSTRACT

We have developed a new steel strip for gaskets that can seal exhaust gas in 900°C environments. The concept for the material specified the seemingly conflicting requirements of high performance through the active use of γ' -Ni₃ (Ti, Al) as a strengthening phase for high-temperature stability, together with a higher melting point for manufacturability. To enhance the relaxation properties, alloying elements that raised the amount and stability of strengthening phase γ' and suppressed the precipitation of the harmful phase η -Ni₃Ti were investigated using thermodynamic calculations, and 5.9 at% of Ti+Al and a ratio of 1.0 of Al/Ti were selected. In addition, to enhance hot rollability, the melting point was increased by reducing Nb and Cr contents as compared to Alloy 718. To maintain initial springiness and the stability of γ' at high temperatures, the cold rolling reduction ratio was set at 30% to 40%. As a result, in heat load tests at 900°C, gaskets made of the developed alloy performed at the same level as Alloy 718 gaskets at 750°C.

1. Introduction

With the goal of mitigating global warming, Japan and other developed nations have declared their aim to realize carbon neutral societies with zero greenhouse gas emissions (net total of emissions and absorption) by 2050⁽¹⁾. Through electrification, the automobile industry is working to bring carbon emissions from driven vehicles to zero. However, there are a large number of issues that need to be addressed before this can become widespread, and it is also important to further develop internal combustion engines, which for now remain the dominant powertrain systems.

Europe is considering expanding the Real Driving Emission (RDE) driving conditions, imposing stricter limits, and enacting other measures under Euro 7, the next round of automobile exhaust gas regulations. In this context, in order to simultaneously enhance thermal efficiency and reduce exhaust gas emissions in internal combustion engines, stoichiometric air-fuel ratio operation is necessary across the full range without enrichment controls, as such controls have the effect of cooling the fuel. The result is that exhaust gas temperatures, which have traditionally been around 900°C, will exceed 1000°C. This will increase the demand for materials that can provide component functionality and durability in these high-temperature environments⁽²⁾. Accordingly, exhaust components need to have high heat

resistance, not only typical exhaust system components such as the turbocharger, catalytic converter, and exhaust pipe, but also the gaskets that are responsible for sealing in the gases where such components are utilized. A gasket is used between the turbocharger and the catalytic converter, where exhaust gas temperatures are about 100°C to 150°C lower than those at the engine outlet. Therefore, if the temperatures of such exhaust gases are expected to increase from 900°C to 1050°C, the gasket needs to provide sealing functionality at 900°C, 150°C more than the former 750°C level.

As shown in Fig. 1, the developed gasket consists of multiple layers of 0.2 mm cold-rolled material formed with ridges (beads). This multi-layered gasket is bolted in place between the flanges of the turbocharger and catalytic converter. The gasket's bead seal mechanism is shown in Fig. 2. During initial installation, the bolting force compresses the beads between the flanges, generating a surface pressure that seals in the exhaust gases.

During driving, heat from the exhaust gas may cause thermal deformation of the flanges, tending to create gaps between the flanges where the bolts are widely spaced. In response, the springiness in the gasket beads makes them conform to the gaps, thus maintaining the surface pressure and the exhaust gas seal. In terms of sealing the gases, therefore, the higher the bead height that remains after

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repeated engine operation (remaining bead), the greater the sealing surface pressure, the better the beads can conform to the gaps in the flanges that may occur during driving, and thus the better the gas sealing functionality.

In order to secure this remaining bead height, it is necessary to not only have high initial springiness (hardness), but also to suppress the relaxation phenomenon that may occur when the seal pressure generated by the flange connection changes over time. Hereinafter, this will be referred as the relaxation properties. Therefore, in order to provide sufficient sealing functionality for this gasket, a material that has reliable relaxation properties at the target temperature of 900°C is required.

As shown in Fig. 3, gaskets are made from steel strips that are manufactured in a series of steps from ingot casting and blooming to hot rolling followed by cold rolling. The strips are then subjected to bead forming and shape punching to produce the desired shape. Typically, any attempt to increase relaxation properties at high temperatures affects the hot and cold rollability, making it is necessary to also consider the product's manufacturability.

Given all of the above, we set out to develop a high-heat-resistant steel strip with reliable relaxation properties at 900°C that also take manufacturability into account.

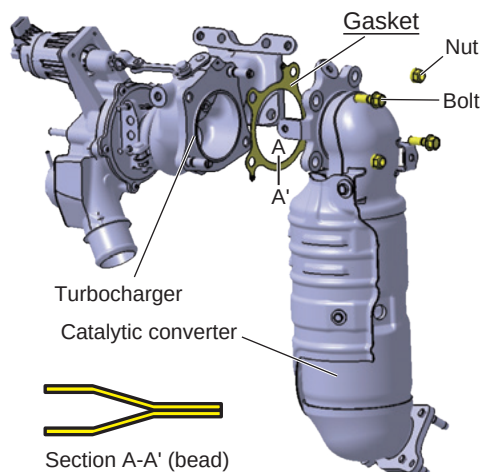


Fig. 1 Gasket in exhaust system

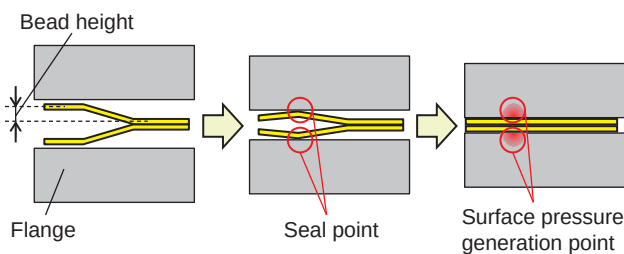


Fig. 2 Assembly flow and sealing mechanism of gasket

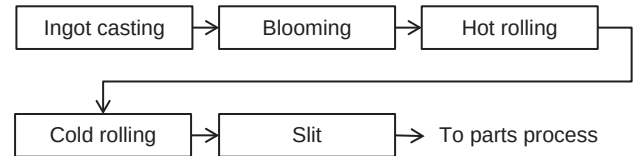


Fig. 3 Outline of steel strip manufacturing process

2. Development Goals and Setting of Target Values

One example of a general-purpose gasket material currently used between exhaust system components is the Ni-based superalloy Alloy 718 (AMS5596). This is a precipitation hardening type alloy, which is one of the alloy-strengthening mechanisms. γ'' -Ni₃Nb, an intermetallic compound of Ni and Nb, and γ' -Ni₃(Al, Ti), an intermetallic compound of Ni, Al, and Ti, are the strengthening phases⁽³⁾. The alloy has reliable relaxation properties under high-stress and high-temperature environments. This alloy is therefore used for gaskets between the turbocharger and catalytic converter in current engines, in which 750°C temperatures can be expected. When this alloy is exposed to temperature ranges above 750°C for certain periods of time, however, one of the strengthening phases, γ'' -Ni₃Nb, transforms into δ -Ni₃Nb, with a different crystal structure⁽⁴⁾, and the alloy's relaxation properties degrade as a result. Therefore, in order to develop a high-heat-resistant strip in which relaxation properties are maintained at the anticipated running temperature increase from 750°C to 900°C that may occur due to future increases in exhaust gas temperatures, it was decided to actively pursue the use of γ' rather than γ'' ⁽⁵⁾ as the strengthening phase, due to its greater thermodynamic stability.

There is concern however that the higher stability of γ' in high-heat-resistance strips may make the hot rolling process shown in Fig. 3 more challenging to carry out. In hot rolling processes that are typically conducted in the temperature range of 700°C to 1200°C, a high-heat-resistant strip exhibits high deformation resistance because of the issue of making γ' particles fully dissolved⁽⁶⁾, and this can lead to cracking and so on. To address this issue and enhance hot-rollability, an existing Ni-based superalloy such as Alloy 263, which has a higher heat resistance than Alloy 718, for example, can lower the solvus temperature of γ' with the addition of Co⁽⁷⁾. However, in recent years, Co is attracting increasing attention for its impact on people and the environment, and so to avoid using Co in this development, the concept of having a high melting point that can expand the upper limit of the hot rolling temperature range was incorporated into the alloy design.

Cold rolling is performed after the strip hot rolling. In cold rolling, increasing the rolling reduction ratio promotes strain hardening (dislocation strengthening) resulting

from accumulated crystal defects (dislocations) in the crystals, producing higher initial springiness. However, introducing excessive dislocations increases the driving force of microstructural changes and is detrimental to high-temperature relaxation properties. Therefore, cold rolling conditions were also assessed. In terms of the performance target of the gasket for which the developed alloy would be used, the goal was to maintain a remaining bead height of 56 μm at 900°C, the same remaining bead height for an Alloy 718 gasket at 750°C, in a heat load test of a single-layer gasket modeled on that mounted between the turbocharger and catalytic converter of a current market vehicle. The processing capacity of a mass-production hot-rolling mill was considered, and manufacturing targets were set to provide a hot working temperature range of 350°C or higher in a Gleeble test that simulates hot-rollability.

3. Alloy Design

In order to achieve the performance and manufacturing targets, thermodynamic calculations were performed to determine the optimal amount of alloying element that would enhance the high-temperature stability of γ' and increase the melting point of the alloy, starting with a base of the standard composition of Alloy 718, Fe-51Ni-19Cr-5.1Nb-0.5Al-1.0Ti-3Mo (mass%). For this investigation, thermodynamic calculations were performed using Thermo-Calc with ThermoTech's thermodynamics database Ni8.

3.1. Stabilized γ' Design for Enhanced Functionality

With regard to the role of each alloying element in Ni-based superalloys, Ti, Al, Nb, etc. stabilize strengthening phase γ' (Nb also stabilizes γ'' , however), while Cr, Mo, etc. perform solid solution strengthening of the matrix⁽⁸⁾. One of the goals of this development was to investigate the optimal quantities of Al and Ti needed to stabilize γ' . Based on the standard composition of Alloy 718, the Ti/Al ratio was fixed at 1.0 atomic percent, and the relationship between the γ' mole fraction and solvus temperature was plotted for varying amounts of Ti+Al. The results are shown in Fig. 4.

As the amount of Ti+Al increases, the γ' solvus temperature rises, and it is necessary to add Ti+Al of 3 at% or more to stabilize γ' at 900°C. On the other hand, the concern exists that if too much Ti+Al is added, the hot workability will degrade with the rise in the solvus temperature. In existing precipitation-hardened-wrought Ni-based superalloys used at 700°C or higher, the amount of precipitation of γ' is set at between 20% and 30%⁽⁹⁾. Accordingly, in this development, the amount of Al+Ti was set at close to 6 at%, with the aim of achieving 25% of γ' .

In addition to the total amount of Ti+Al, the Ti/Al ratio is also an important parameter for the stability of γ' ⁽¹⁰⁾. The ratio value has an impact not only on γ' , but also on the stability of the Ni and Ti intermetallic compound,

$\eta\text{-Ni}_3\text{Ti}$. Typically, in Ni-based superalloys η precipitates in a plate shape and reduces high-temperature strength⁽¹¹⁾. For this reason, it is preferable to have a design that avoids precipitation. Therefore, the mole fractions of γ' and η were calculated at 900°C against the Ti/Al ratio at a fixed Ti+Al amount of 6 at% (Fig. 5). Accordingly, the amount of γ' increases as the Ti/Al ratio increases, up to around Ti/Al = 1. Above this, however, γ' starts to decrease and η is formed and begins to increase. Based on these calculations and a consideration of the balance between the amounts of γ' and η , the Ti/Al ratio was set at about 1.

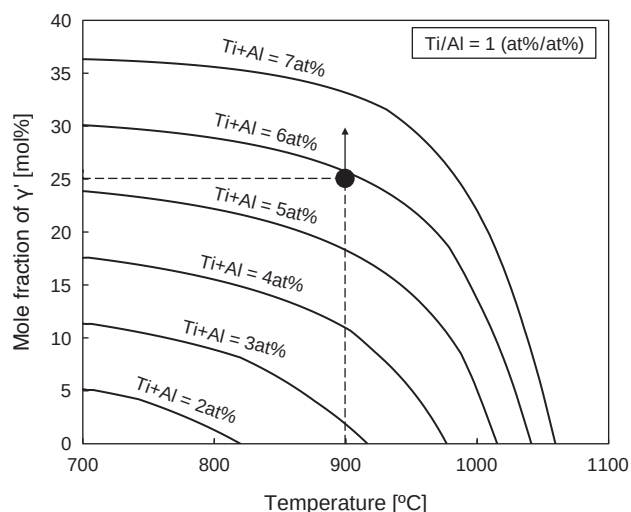


Fig. 4 Relationship between temperature and γ' mole fraction of Fe-51Ni-19Cr-5.1Nb-3Mo-Al-Ti (mass%) with various Ti+Al contents (at%)

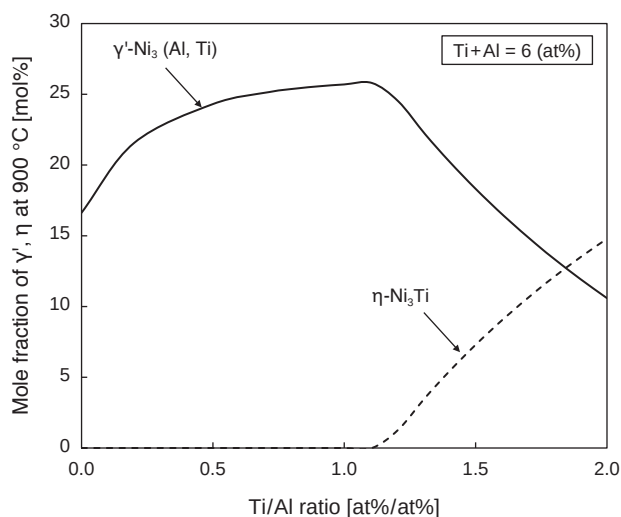


Fig. 5 Relationship between Ti/Al ratio (at%/at%) and γ'/η mole fraction at 900°C of Fe-51Ni-19Cr-5.1Nb-3Mo-Al-Ti (mass%)

Table 1 Chemical compositions of developed alloy and Alloy 718

	Chemical composition [mass%]							Ti+Al [at%]	Ti/Al [at%/at%]
	Ni	Cr	Fe	Nb	Al	Ti	Mo		
Developed alloy	50	15	Balance	1.3	1.4	2.5	1	5.9	1.0
Alloy 718	51	19	Balance	5.1	0.5	1.0	3	2.3	1.1

3.2. Higher Melting Point Design to Enhance Hot Workability

As discussed in Chapter 2, due to the concern that stabilizing γ' decreases hot workability, it was decided to enhance ductility at high temperatures above 1000°C, at which γ' can be fully dissolved, to secure a sufficient hot working temperature range. Since high-temperature embrittlement has a major impact on the decrease in ductility at high temperatures, a study was conducted to reduce the amount of elements in order to increase the melting point, with which this is correlated. With regard to the alloying element groups, as Ni, Al, and Ti constitute γ' and thereby impact performance, the focus was turned to Nb and Cr, other elements that are highly sensitive to melting point effects. Nb was studied first, and a phase diagram was calculated based on an Alloy 718 component with the amount of Nb on the horizontal axis (Fig. 6). This showed that for every 1 mass% reduction in the amount of Nb added, the melting point (solidus temperature) rose about 28°C. In Alloy 718, Nb is the alloying element that stabilizes γ'' , which is the main strengthening phase of the alloy. In this development, however, γ' was being used instead as the main strengthening phase, and so reducing the amount of Nb was expected to have only a small effect on the alloy's relaxation properties. However, a certain amount

of Nb was considered necessary, because greatly decreasing the amount of Nb has been shown to decrease the stability of γ' and the solvus temperature of γ' (γ' - solvus). Therefore, to help prevent the formation of harmful phase δ at the anticipated service temperature of 750°C to 900°C for the alloy being developed, Nb was reduced to 1.3 mass%, where the δ solvus temperature (δ - solvus) is below 750°C.

Next, Fig. 7 shows the calculated phase diagram with the amount of Cr on the horizontal axis. This shows that for Cr, as well, each additional 1 mass% results in an approximately 10°C rise in the melting point, and so the amount of Cr was reduced to 15 mass%, still within a range that did not degrade oxidation resistance.

3.3. Composition of Developed Alloy

Table 1 shows the developed alloy composition, taking into consideration performance and manufacturability, and as based on preceding Sections 3.1 and 3.2. The Mo element contributes to solid solution strengthening, which is one of the strengthening mechanisms for alloys. However, because precipitation hardening of γ' was mainly being used in the developed alloy, and Mo is a rare metal and has presented an increasing resource risk in recent years, the amount was reduced compared to Alloy 718. The solvus temperature and melting point of γ' in the developed alloy are 994°C and

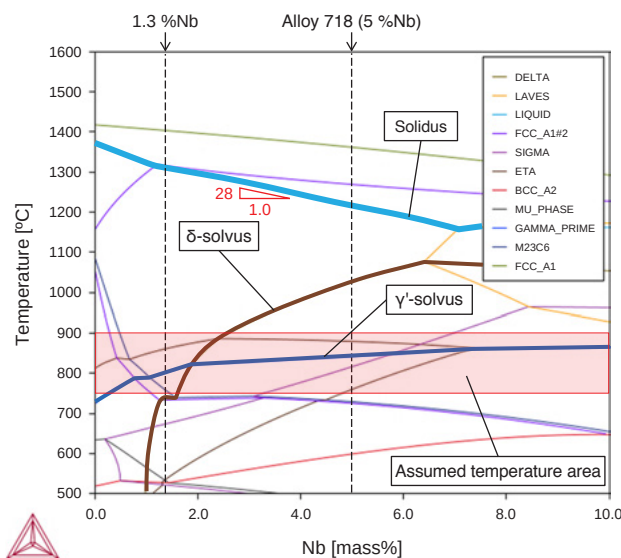


Fig. 6 Calculated vertical section of Fe-51Ni-19Cr-3Mo-0.5Al-1Ti-Nb (mass%)

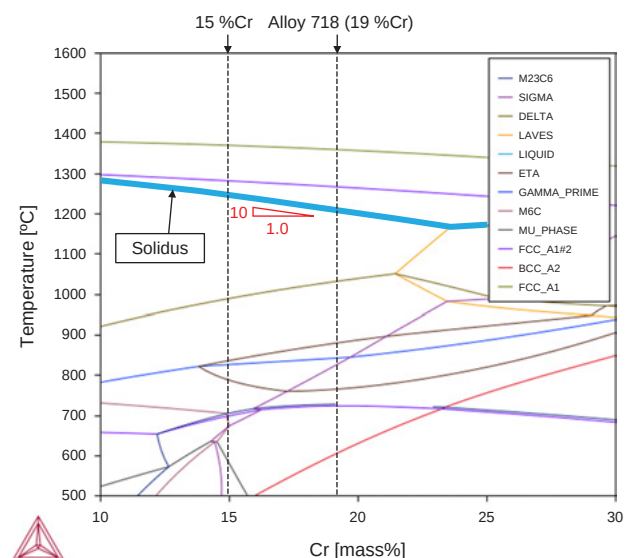


Fig. 7 Calculated vertical section of Fe-51Ni-3Mo-0.5Al-1Ti-5.1Nb-Cr (mass%)

1292°C, respectively, as calculated by Thermo-Calc. Both parameters are higher than the 835°C and 1204°C figures for Alloy 718, confirming that the component composition meets both performance and hot workability considerations.

4. Cold Rolling Condition Setting

As described in Chapter 2, in consideration of initial springiness, it is preferable to increase hardness with the highest possible cold rolling reduction ratio. However, excessive cold rolling promotes the precipitation of η , reducing the high-temperature strength of the alloy⁽¹²⁾. Since this, in turn, prompts concerns about reduced relaxation properties, it is necessary to clarify the cold rolling reduction range within which there is no effect on the stability of γ' , and to establish an upper value. For this study, the compression ratio of prismatic test pieces stood in for the cold rolling reduction ratio, and the samples were created as shown in Fig. 8 for heat exposure tests. The stability of γ' was subsequently assessed, based on the hardness and microstructural observation, and the cold rolling reduction ratio established.

For the test method, compressed samples were first prepared at various compression ratios. They were then exposed to high temperatures in a muffle furnace for extended periods of time, before being cooled in water. The target temperature exposure was 900°C, and the

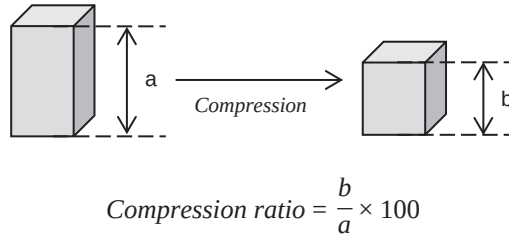


Fig. 8 Preparation method of compressed sample

time was set at 50 hours, sufficient for high-temperature relaxation to stabilize⁽¹³⁾. A Vickers hardness test was then used to measure five points in the center cross-section of the samples at each compression ratio. The structure was examined for the presence of γ' and η with a scanning electron microscope (SEM). Figure 9 plots the hardness after heat exposure against the compression ratio of the prismatic test pieces. The plots are the averages, and the upper and lower lines respectively show the maximum and minimum values for each temperature. The results showed that the hardness increased due to strain hardening as the compression ratio increased, but at over 40%, hardness after thermal exposure decreased, while the variation of hardness increased greatly. To analyze the reason for, the state of precipitation of γ' and η were observed with an SEM in samples with 0% and 50% compression ratios (Fig. 10).

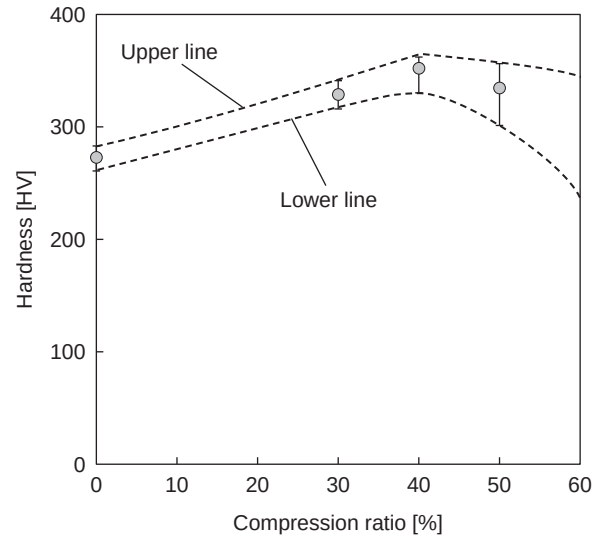


Fig. 9 Relationship between compression ratio of developed alloy and hardness after exposure at 900°C for 50 hours

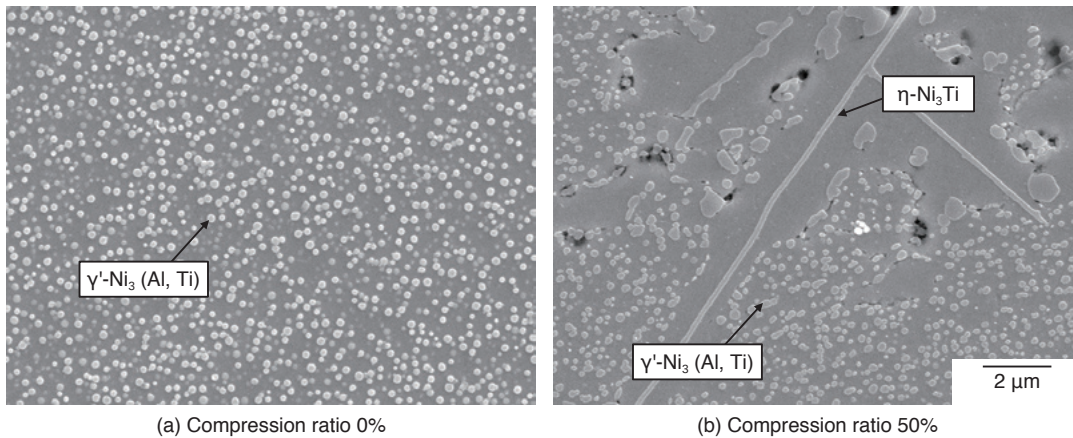


Fig. 10 Secondary electron images of developed alloy after compression and exposure at 900°C for 50 hours

In the sample with 0% compression ratio, homogeneous distribution of fine γ' particles within the grain interior were observed, while in that with 50% compression ratio, needle-like η particles were observed in addition to the γ' particles. In addition, areas with no γ' particle precipitation were confirmed in areas close to the needle-like η particles.

These results suggest that the reason that hardness decreases when the compression ratio exceeds 40% is the occurrence of areas without γ' precipitation, and the reason for the increased variation in hardness is the variation in the structure, which includes mixed areas of γ' precipitation and γ' non-precipitation. Based on these results, the cold rolling reduction ratio was set at between 30% and 40%, to secure the stability of γ' while taking into account initial springiness.

5. High-temperature Property Test Method and Results

Up to Chapter 4, it was confirmed that the examined alloy composition and cold rolling reduction ratio satisfied the development targets for performance and manufacturing. With regard to performance, the characteristics of the developed alloy and Alloy 718 were compared with a relaxation test using standard round bar test pieces.

5.1. Relaxation Assessment Testing

The alloy configured in Section 3.3 was hot forged into round bars and solution treated at the assumed annealing temperature during cold rolling. The round bars were then cold swaged to introduce a plastic strain equivalent to 30% to 40%. This was followed by heat treatment for 50 hours at each of the test temperatures described below to produce round bar test pieces as depicted in Fig. 11.

The relaxation tests were conducted in accordance with ASTM E328-13⁽¹⁴⁾. Change in stress over time was recorded for up to a maximum of 50 hours with a gauge length of 12 mm and a tensile strain of 1%. The test temperatures were 800°C, 850°C, and 900°C, and the same tests were performed on Alloy 718 for comparison.

The results of the relaxation testing are shown in Fig. 12. At 900°C, the residual stress of Alloy 718 was zero, while that of the developed alloy remained at about 35 MPa. This confirmed that the developed material has better high-temperature relaxation properties than Alloy

718. The enhancement in the residual stress in the high-temperature range is presumed to be due to γ' remaining stable as a strengthening phase up until 900°C, which suppresses stress relaxation at high temperatures, as per the development concept.

5.2. Assessment Test for Hot-rollability

The test pieces were made from round bars hot forged in the same ways as the pieces in Section 5.1, then machined into parallel lengths of 20 mm with a diameter of 4.5 mm. First, the test pieces were placed inside a Gleeble testing machine and heated for 100 seconds until reaching 1150°C, at which temperature γ' particles completely dissolved, then held for 60 seconds. Next, the test pieces were cooled or heated to the test temperature (from 900°C to 1200°C) at a rate of 10°C/s, and held for 60 seconds. They were then subjected to a tensile test with a crosshead speed of 50.8 mm/s. The evaluation metric was established as the temperature range at which ruptures may occur with 50% or higher reduction of area. This temperature range is the range where hot rolling can be performed⁽¹⁵⁾. The results were then compared to Alloy 718 (Fig. 13). In addition, the temperature dependence of the γ' mole fraction and liquid phase of the developed alloy and Alloy 718 were calculated using Thermo-Calc (Fig. 14).

Figure 13 shows that the developed alloy has a lower reduction of area from the low-temperature side up to 950°C, but has a higher value in higher temperature ranges. The reason for this may be seen in Fig. 14, which shows that the developed alloy has a higher γ' mole fraction at the low-temperature end compared to Alloy 718. This means that during the test γ' precipitates more easily, decreasing ductility. At the high-temperature end, however, the melting point, which corresponds to the start of high-temperature

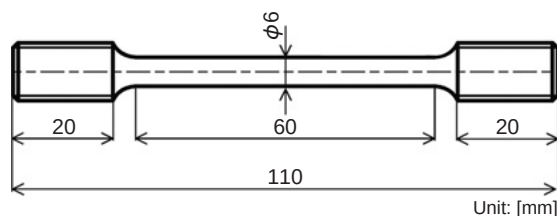


Fig. 11 Sample shape for relaxation test

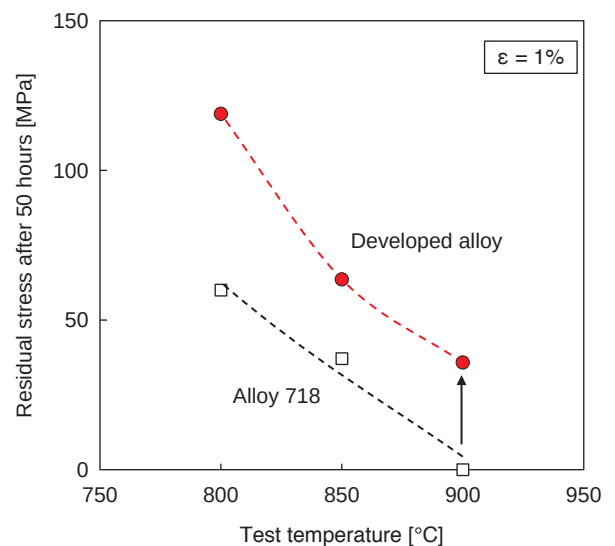


Fig. 12 Relaxation properties of developed alloy and Alloy 718 using round bar test pieces

embrittlement, is higher, and this is assumed to maintain ductility.

It follows from the above then that the developed alloy has, compared to Alloy 718, a higher temperature range in which hot rolling is possible, in line with the design intent of the higher melting point. These results confirm that the temperature range at which hot rolling is possible is approximately 450°C, and that the development target of 350°C or higher has been achieved.

5.3. Gasket Heat Load Test

Using equipment that could be expected to be used in mass production, prototypes of the developed strips were

created using the alloy composition and cold rolling ratio configured in Chapters 3 and 4. Gaskets with a bead height of 0.45 mm were fabricated from both the prototype strips and Alloy 718 strips. As shown in Fig. 15, the gaskets were mounted between flat jigs made of SUS303, 20 mm thick with M10 holes, secured with bolts, and exposed to the air at specified temperatures for 50 hours.

After the test ended, the remaining bead height was assessed and compared to the Alloy 718 gasket. The bead measurement location was an area in which the distance between bolts was large (Area A), an area that is more challenging to seal. Figure 16 shows the results.

At 900°C, the remaining bead height for the Alloy 718 gasket was 0 μm , while that of the developed alloy was 64 μm . This was presumed to be because strengthening phase γ' in the developed alloy continued to exist stably up to 900°C, thus providing confirmation of the development concept. The results showed that the remaining bead height of the developed alloy at 900°C exceeded the 56 μm remaining bead height of Alloy 718 at 750°C, confirming that the developed target had been achieved.

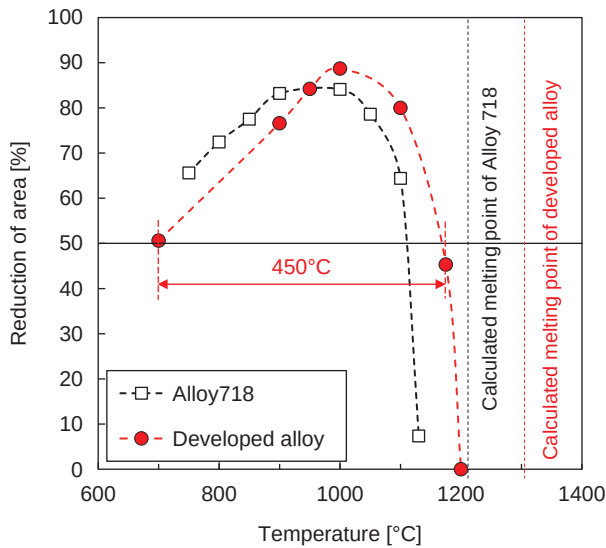


Fig. 13 Result of Gleeble test for developed alloy and Alloy 718

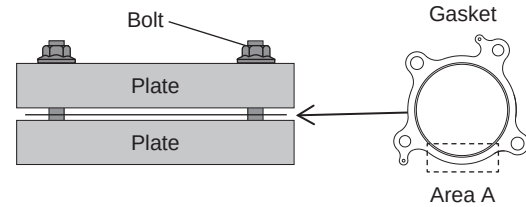


Fig. 15 Layout of gasket test and evaluated bead area after heat exposure

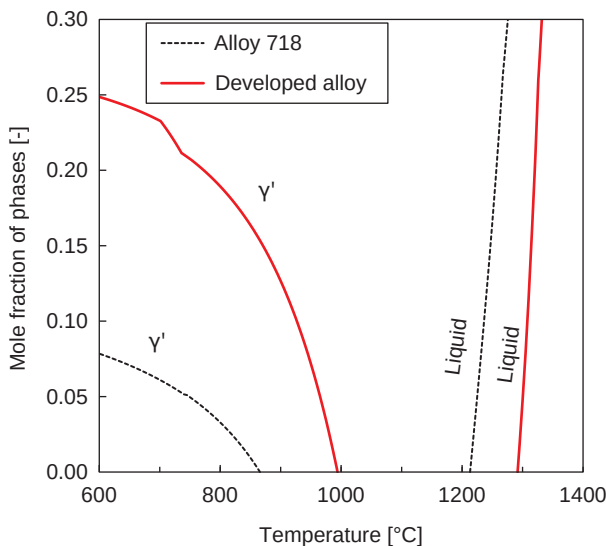


Fig. 14 Calculated mole fraction of γ' and liquid phases for developed alloy and Alloy 718

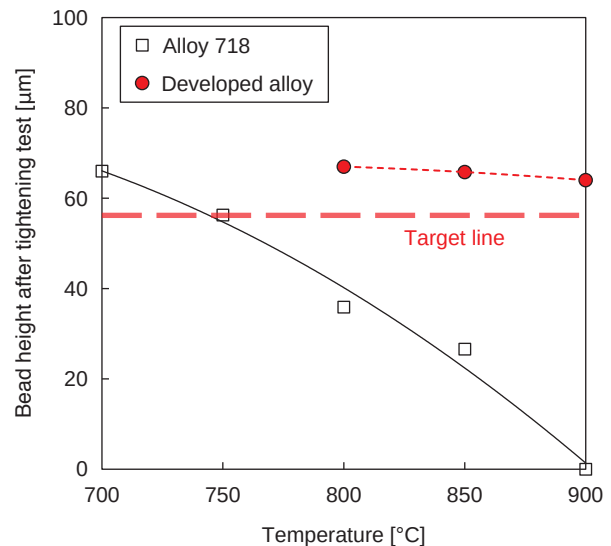


Fig. 16 Result of tightening test after exposure at each temperature

6. Conclusions

In order to develop a strip for gaskets that can provide sealing up to 900°C as required for compliance with future exhaust gas regulations, we have developed a Co-free strip, based on the composition of Alloy 718, that makes active use of γ' as the strengthening phase.

- (1) From calculations using Thermo-Calc, setting Ti+Al at 5.9 at% and the Al/Ti ratio to 1.0 (at%/at%) maximized the amount of γ' , which enhanced relaxation properties and suppressed the precipitation of harmful phase η , which degrades said properties.
- (2) At the same time, based on Thermo-Calc melting point calculations, the amounts of Nb and Cr were reduced compared to Alloy 718 to 1.3 mass% and 15 mass%, respectively, thereby raising the melting point and helping to ensure the possibility of hot rolling at temperatures at or above 450°C.
- (3) In consideration of the initial spring properties and stability of γ' during use, the cold rolling reduction ratio was set from 30% to 40% in order to suppress precipitation of η and the formation of areas without γ' precipitation at 900°C.
- (4) In heat load tests, it was confirmed that gaskets made of the developed alloy and manufactured with equipment expected to be used in mass production, maintained a remaining bead height at 900°C that was equivalent to the remaining bead height at 750 °C of a gasket made of Alloy 718.

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