Technical Report

Evaluation Technology of Heat Resistant Gasket and Application of NSSC™ 302BN

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Abstract

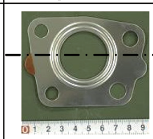
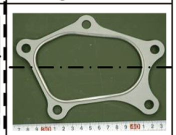
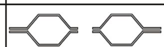
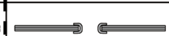
In response to environmental issues, exhaust gas purification systems have been introduced in the automobile field. Along with its introduction, the application range of heat resistant gaskets for connecting parts has been expanding. We developed a new gasket evaluation method for the purpose of applying it to gaskets used under intermediate temperature environments, and various dominant factors which affect the gasket characteristics were evaluated. It became evident that the gas leakage amount used as an important indicator to understand the gasket characteristics was reduced by reducing the amount of high temperature degradation, and suppressing the deformation of the bead head. As a result of an evaluation of the leak resistance after the sample was heated at 700°C, the NSSC 302BN (20Cr-11Ni-1.7Si-Nb-N-REM) indicated excellent results. The NSSC 302BN is being applied practically as a gasket material to be used in the intermediate temperature range.

1. Introduction

In recent years, regulations have been strengthened to alleviate air pollution and global warming that have worsened with the worldwide proliferation of automobiles. The nitrogen oxides (NOx) and particulate matter (PM) contained in the automobile exhaust gases are harmful substances to the human body. Automobile exhaust gas regulations have been tightened.[1, 2] At the same time, calls have been rising for improving automotive fuel efficiency and reducing automotive CO_2 emissions to combat global warming.[3]

To comply with these exhaust gas regulations, automobile manufacturers have been developing new technologies and additional parts. For example, increasing numbers of diesel cars have been fitted with exhaust gas recirculation (EGR) systems for reducing the NOx emissions and with turbochargers for improving the fuel efficiency.[2, 4] These changes have been increasing the use of flanges and metal seals (hereinafter collectively referred to as gaskets) required to connect parts. Because automotive gaskets are exposed to exhaust gases of about 850 to 1050°C, they must resist heat. Because cylinder head gaskets and exhaust manifold gaskets that contact engine blocks are cooled during use, they must have strength rather than heat resistance.

Figure 1 shows the appearance, structure, and materials of gaskets that were recovered from the market and investigated. For a low temperature range of up 500°C, one or two full bead gaskets are

	Low temperature range (~500°C)	Medium temperature range (500~700°C)	High temperature range (700°C~)
Structure			
Cross section	 Full bead	 Half bead	 Grommet
Number of layers	1~2	~4	~6
Applicable material	SUS301 NSSC 431DP-2 (α+M phase)	γ-SUS (high N, Mn)	NCF625 (γ´ phase)

⬚ : target

Fig. 1 External appearance and structure of various gaskets and example of applicable material

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used per location and their material is mainly the metastable austenitic steel SUS301 (17Cr-7Ni) HT.[5] The NSSC 431DP-2 (16.5Cr-2Ni)[6] with a ferrite-martensite duplex microstructure is used as a material for gaskets in some car models. Strength is emphasized for gaskets for use in the low temperature range. For a medium-temperature range of 500 to 700°C, four or less half-bead gaskets are used per location. Their materials are high-N and -Mn austenitic stainless steels.[7, 8] For gaskets in the medium-temperature range, high-temperature strength during heating rather than at room temperature is emphasized. For a high temperature range of 700°C and above, six or less grommet gaskets are used per location. Their material is the NCF625 (22Cr-9Mo-4Nb-0.2Ti-0.2Al-Ni), a nickel-based alloy strengthened by the precipitation of the γ' phase. Heat resistance is stressed for gaskets in the high-temperature range. In this study, we targeted gaskets in the medium-temperature range of 500 to 700°C for the purpose of applying heat-resistant stainless steels.

Nippon Steel Stainless Steel Corporation developed a new gasket evaluation method to assess the properties of gaskets in a medium-temperature environment. Conventionally, the main property required of gasket materials was strength to ensure permanent set resistance. We established the laboratory evaluation technology to study the leak resistance of gaskets in terms of the material type, shape, and surface morphology. We investigated the properties required of gaskets with our evaluation method and proposed a new gasket material. In this paper, we introduce the evaluation method and the NSSC 302BN (20Cr-11Ni-1.7Si-Nb-N-REM)[9], a new gasket material we proposed according to our study with our new evaluation method.

2. Experimental Materials and Methods

2.1 Materials

Table 1 shows the chemical composition of the experimental materials. The NSSC 302BN and NSSC S-4 (17Cr-15Mn-N) with high-temperature strength similar to that of conventional gasket materials and the NSSC ER-1 (19Cr-13Ni-3.3Si-Nb) with high-temperature oxidation resistance were selected as candidate materials. The SUS301 used now as low-temperature gasket material and the NSSC 431DP-2 with softening resistance up to 500°C and like that of the SUS301 were used as comparative materials. The NSSC 302BN was used in the annealed and rolled conditions to confirm the effects of its properties. The NSSC 431DP-2 was used as heat treated in the duplex microstructure, a condition conventionally employed in the gasket application. The other steels were used in the rolled condition with a reduction ratio of 40 to 60%.

2.2 Methods

Figure 2 shows the evaluation method for gasket properties. The permanent set amount that indicates the deformation of beads and the leak amount that indicates gas leakage from the gasket seal were measured as evaluation indexes. The effect of the gasket shape and the effect of the residual bead height (tightening force) were examined as influential factors in the gasket evaluation. The preliminary conditions were set as follows. Ring-shaped flat sheet specimens with a thickness of 0.25 mm, an outer diameter of 50 mm, and an inner diameter of 30 mm were beaded with a full, mountain-shaped bead with a height of about 500 μm. The formed gasket specimen was set between two restraining plates (made of the SUS310S and 15 mm thick) and was clamped with four M10 SUS304 bolts by tightening the bolts with a torque of 15 N·m each. To equalize the tightening force, ring-shaped spacers (made of the SUS310S and 0.4 mm thick) for clearance adjustment were inserted into the tightening bolts. The tightening rate was set to 70%. The gasket specimens were then leak tested in the bolt-clamped condition to determine the effect of tightening at room temperature. To understand the effect of heating, some gasket specimens were leak tested after heating at 700°C for 120 h. The heating temperature was set to the upper limit

Table 1 Chemical composition of experimental materials

(mass%)

	C	Si	Mn	Ni	Cr	N	Nb	REM
NSSC 302BN	0.06	1.7	1.1	11.0	20.0	0.15	0.1	Addition
NSSC ER-1	0.04	3.3	0.8	13.1	18.7	0.01	0.1	–
NSSC S-4	0.17	0.4	14.6	1.3	17.3	0.44	–	–
SUS301	0.10	0.7	0.9	6.9	16.9	0.06	–	–
NSSC 431DP-2	0.06	0.5	0.3	1.9	16.3	0.01	–	–

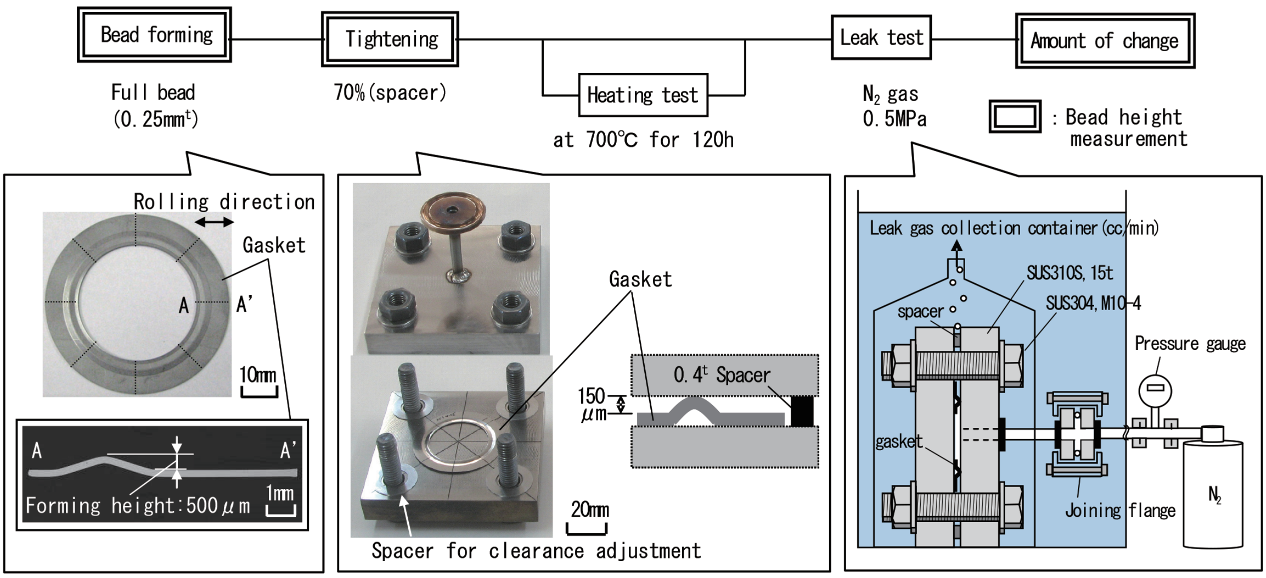

Fig. 2 Evaluation method for gasket properties

temperature for gaskets for use in the medium-temperature range.

The gasket specimens were leak tested by immersing them in water together with their restraining jig. The gasket specimens to evaluate the effect of 700°C heating were cooled to room temperature and then leak tested. Nitrogen gas was used as the gas for measuring the leak amount. The leak amount (cc/min) was determined by collecting the bubbles leaking from the gasket seal when the nitrogen gas was introduced at a pressure of 0.5 MPa. After that, the gasket was removed from the restraint jig and its height was measured at eight circumferential positions with a micrometer. The bead height was measured by removing the jig after beading as well as after bolt tightening and heating with the jigs removed. The permanent set amount from beading to bolt tightening with the restraining plates was defined as the room-temperature permanent set amount. The permanent set amount from bolt tightening to heating at 700°C was defined as the high-temperature permanent set amount.

3. Results and Discussion

3.1 Permanent set resistance and leak resistance of stainless steels

The specimen heating temperature and time were set to 700°C and 120 h, respectively. **Figure 3** shows an example of the residual bead height of the specimens measured after the respective steps. Three austenitic stainless steels with similar high-temperature strength were used as evaluation materials. The SUS301 and NSSC 431DP-2 were used as comparative materials. The initial beads were formed to a height of about 500 μm. The bead height then changed with springback. The higher the room-temperature strength of the steel, the lower the bead height became and was about 400 to 450 μm. The bead height after bolt tightening was about 200 to 250 μm and was larger than the spacer height. It was the largest for the SUS301. The formation of the martensite phase in the SUS301 increases its room-temperature strength. The SUS301 beads are difficult to deform when clamped and their height is the largest. The bead height after heating at 700°C for 120 h is about 150 μm and is close to the spacer height. This bead height is the lowest for the SUS301. The decomposition of the deformation microstructure and martensite phase makes the bead height the lowest for the SUS301. For the three austenitic stainless steels, the bead height decreases in the order of the NSSC S-4, NSSC 302BN, and NSSC ER-1. It may be speculated from these results that the high-temperature strength of the experimental steels can be approximately arranged by the N content.

Figure 4 shows the measurement results of the permanent set amount of each steel. The permanent set amount is the difference in the bead height between the test steps. The smaller the change in the permanent set amount between the test steps, the higher the permanent set resistance. The room-temperature permanent set amount is larger than the high-temperature permanent set amount for each steel. **Figure 5** shows the measurement results of the leak amount between the test steps. The leak amount after bolt tightening at room temperature was 50 cc/min or less and is not significantly different for all the experimental steels. The leak amount after heating at 700°C for 120 h was larger than that after bolt tightening and was the largest for the NSSC 431DP-2. According to these results, the NSSC 431DP-2 with the largest leak amount and the NSSC S-4 with a smaller high-temperature permanent set amount than the NSSC 302BN but a larger leak amount were selected and compared.

Figure 6 shows the bead top shape after heating at 700°C. In the 3D bead top shape, the flat region (white dot bordered region) where the top height is within 7 μm of the maximum height is wider for the NSSC 431DP-2 than for the NSSC S-4. That is, the bead top deformation is larger for the NSSC 431DP-2. This deformation is consid-

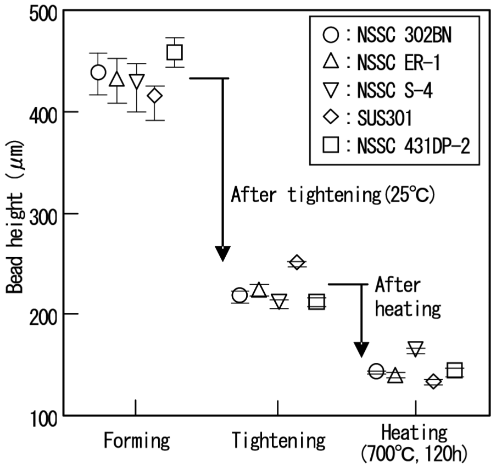

Fig. 3 Height of residual bead after each process

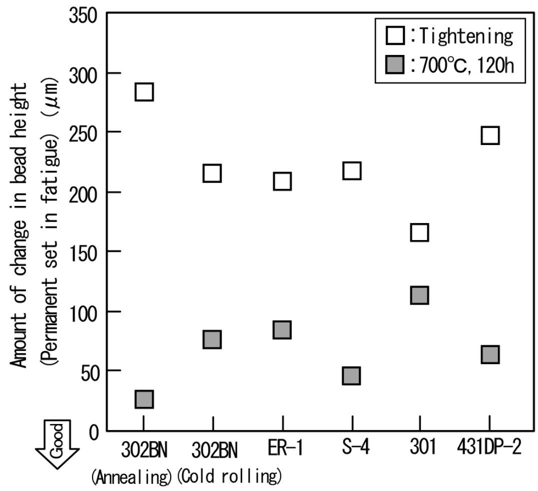

Fig. 4 Measurement results of permanent set amount of each steel

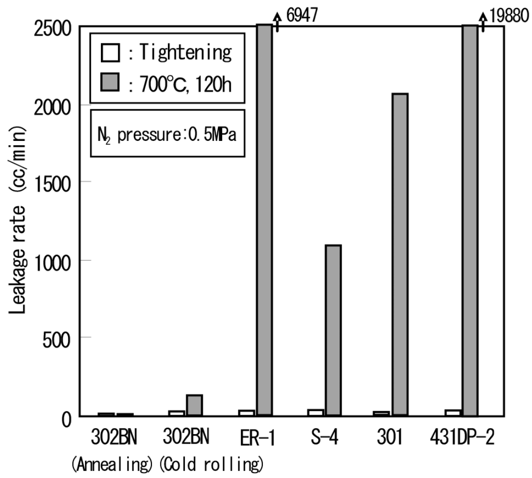

Fig. 5 Measurement results of leakage amount between each process

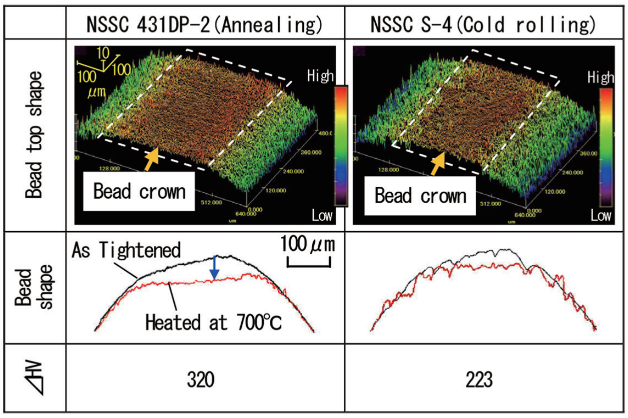

Fig. 6 Bead head shape after heated at 700°C
(ΔHV: material hardness-hardness after heating)

ered to have resulted from the decrease in the steel strength with heating. Although the details will be described later, the bead top deformation increased as the difference in ΔHV (or the difference between the initial material hardness and the material hardness after heating at 700°C for 120 h). The bead shape measured with a stylus surface profilometer is also shown in Fig. 6. As indicated by the arrow, the heating-induced permanent set is larger for the NSSC 431DP-2 than for the NSSC S-4. The surface roughening of the bead top with the ductility drop that is estimated to have been caused by cold rolling is observed for the NSSC S-4. This reason shows that an annealed steel with high high-temperature strength is a suitable gasket material. For actual gaskets, the initial bolt tightening force when installing the gasket is important. For gaskets installed with a large bolt tightening force, annealed steels may not be strong enough. According to the above screening results, the rolled NSSC 302BN with both permanent set resistance and leak resistance was selected as the recommended material.

3.2 Effect of material factors on leak resistance

To clarify the gasket materials applicable in the medium-temperature range, it is important to understand the effect of various controlling factors on the gasket properties.

In the previous section, we described that the leak amount is affected by the high-temperature permanent set amount and by the bead top deformation and surface roughness. We also noted that the high-temperature permanent set amount cannot always be arranged by the high-temperature strength and the room-temperature hardness after aging. In this section, we study the effects of work hardening and grain size to clarify the material factors. The evaluation material was limited to the NSSC 302BN.

3.2.1 Effect of work hardening (annealed steel and rolled steel with a reduction of 20% or 40%)

The effect of work rolling was evaluated by using the NSSC 302BN specimens finish rolled with a reduction of 20% or 40%. For reference, the NSSC 302BN specimens annealed but not rolled were added to the experimental materials. **Figure 7** shows the microstructure and surface hardness of the NSSC 302BN specimens rolled by changing the reduction ratio. Elongated grains were observed in the NSSC 302BN specimen rolled with a reduction of 40%. The surface hardness was HV218, HV329, and HV393 for the NSSC 302BN specimens annealed (but not rolled), rolled with a reduction of 20%, and rolled with a reduction 40%, respectively.

Figure 8 shows the relationship between the high-temperature permanent set amount after heating at 700°C for 120 h and the leak amount. A correlation was observed between the high-temperature permanent set amount and the leak amount. The specimens rolled with a lower reduction ratio had a smaller high-temperature permanent set amount and had a higher leakage resistance. This suggests that if the room-temperature strength is increased by rolling before gasket forming, the leak resistance of the medium-temperature gaskets does not improve but rather worsens.

Figure 9 shows the relationship between the ΔHV after heating at 700°C for 120 h and the high-temperature permanent set amount.

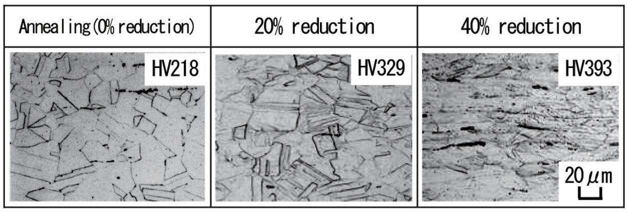

Fig. 7 Metallographic structure and surface hardness of NSSC 302BN with varying rolling ratio

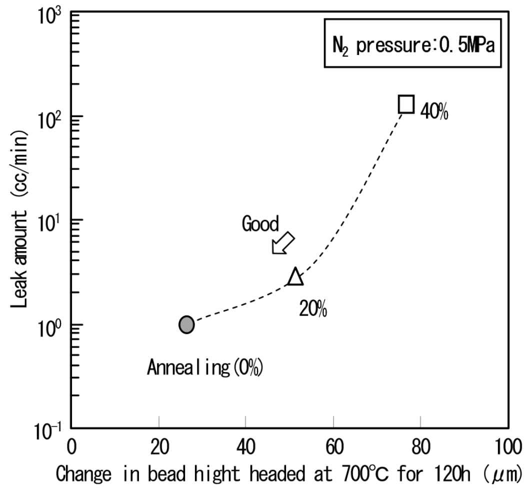

Fig. 8 Relationship between high-temperature permanent set amount after heating at 700°C for 120 h and leak amount

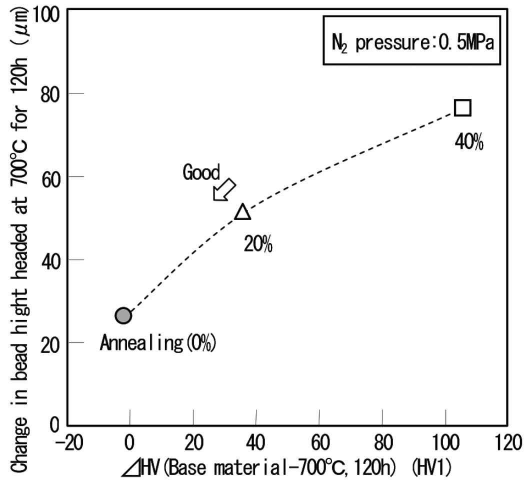

Fig. 9 Relationship between ΔHV after heating at 700°C for 120 h and high-temperature permanent set amount

The ΔHV is the difference between the room-temperature hardness of the specimens and the room-temperature hardness of the specimens after heating at 700°C and 120 h. The smaller the hardness difference before and after heating, the smaller the high-temperature permanent set amount tends to be. The relationship shown in Fig. 8 reveals that the hardness difference before and after heating greatly affects the leak amount.

3.2.2 Effect of grain size before rolling

The finish rolling ratio was set to 40% for the evaluation specimens. The effect of the grain size was evaluated by using specimens with two different grain sizes. **Figure 10** shows the inverse pole figure (IPF) maps and surface hardness of the finished rolled specimens. The annealing conditions were adjusted to make the initial strength as uniform as possible. The average grain size and the surface hardness were set to 6.6 μm and HV393 for the fine-grained specimens, respectively, and to 2.5 μm and HV401 for the ultrafine-grained specimens, respectively.

Table 2 shows the hardness change before and after heating at 700°C for 120 h and the gasket properties. The ΔHV after heating at 700°C for 120 h was HV15 smaller for the ultrafine-grained specimens with higher strength, but the leak amount was larger. In other words, the ultrafine-grained specimens were presumably more likely to decrease in strength when heated, but it became evident that it was not always possible to arrange the results by the ΔHV. The details are described later. The deformation due to the permanent set of the beads occurred only for the ultrafine-grained specimens of the NSSC 302BN and of the NSSC 431DP-2 shown in Fig. 6. Both steels are characterized by their fine grain size. This suggests that creep deformation is involved in the deformation of the bead top. It is possible that the high-temperature permanent set may have completed at the start of soaking after heating. A creep test was conducted to evaluate the results in a short time. The hardness of the fine-grained and ultrafine-grained specimens was HV393 and HV401, respectively. The creep deformation was evaluated as the effect of the reaction force by assuming that the surface pressure was almost the same. **Figure 11** shows the creep test method, creep elongation, and cross-sectional microstructures. The specimen was installed in the heating furnace and heated under no load from room temperature to 900°C in 15 min. The creep elongation between the gauge marks and the cross-sectional microstructure at the center were investigated when the specimen was loaded for 2 min with 40 MPa, just below the offset yield strength. The creep elongation was 0.6% for the fine-grained specimen and was a larger 4.4% for the ultrafine-grained specimen. The rolling ratio was unified to 40% for both the fine-grained and ultrafine-grained specimens. Because grain growth was confirmed not to have occurred during the creep test, the effect of the grain size before rolling is suggested to have been involved in the bead top deformation.

3.3 Effect of surface shape before and after heating at 700°C

Figure 12 shows the appearance and shape of the bead top before and after heating at 700°C under the effects of work hardening and grain size. The surface roughening of the bead top after bead tightening is larger for the specimens with a larger grain size. This surface roughening of the bead top is presumed to affect the leak amount after room-temperature tightening. The surface roughening is retained after heating at 700°C for 120 h. The depression and spread of the bead top before and after heating at 700°C are conspicuous on the specimens with a higher rolling ratio and a smaller grain size. It is presumed that the leak amount after heating increased.

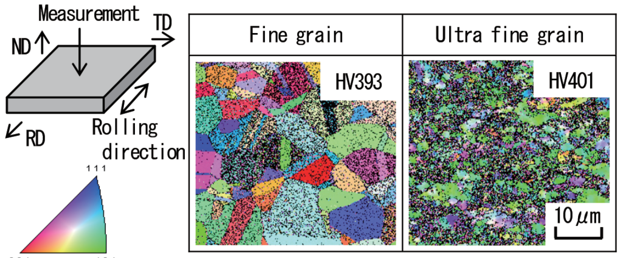

Fig. 10 Inverse pole figure map (IPF map) and surface hardness of finished rolled material

Table 2 Hardness change before and after heated at 700°C for 120 h and gasket characteristics

Grain	Grain size	HV (material)	HV (700°C, 120 h)	ΔHV	Leakage rate
Fine	6.6	393	287	106	126
Ultra Fine	2.5	401	310	91	≥10000

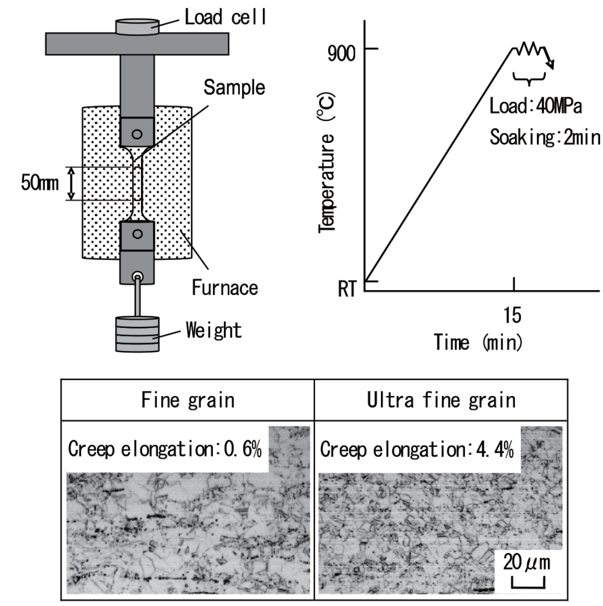

Fig. 11 Creep test method, creep elongation, and cross-sectional microstructures

3.4 Controlling factors of gasket properties

From the above results, it is possible that the effect of the crystal grain size was involved in the balance between the surface pressure applied to the bead during room-temperature tightening and the resultant reaction force during heating. These actions are considered below.

Figure 13 schematically illustrates the surface pressure and reaction force of a high work hardening specimen and an ultrafine-grained specimen as high-strength specimens and a low work hardening specimen (annealed specimen) and a fine-grained specimen as low-strength specimens in the 700°C heating evaluation.

It became evident that the leak resistance cannot always be accurately evaluated by the conventional methods of evaluating the high-temperature strength and hardness after heating. The reason is

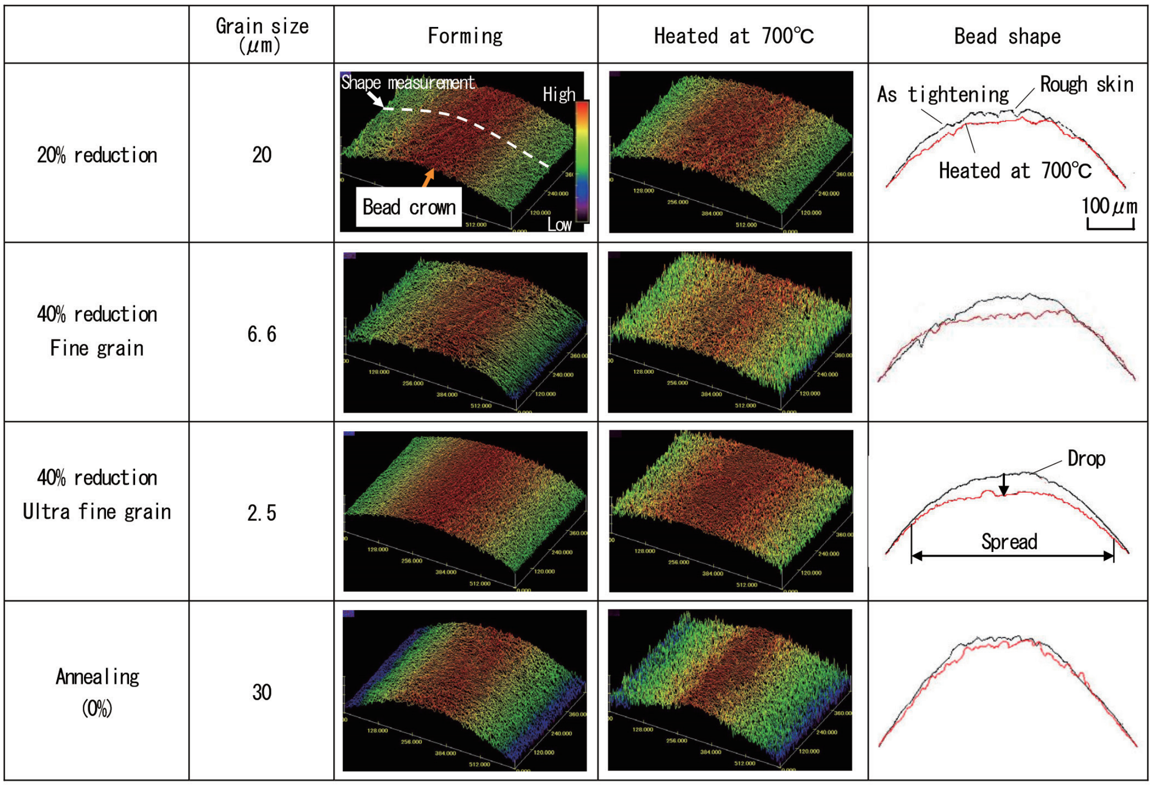

Fig. 12 Bead head shape before and after heated at 700°C for 120 h

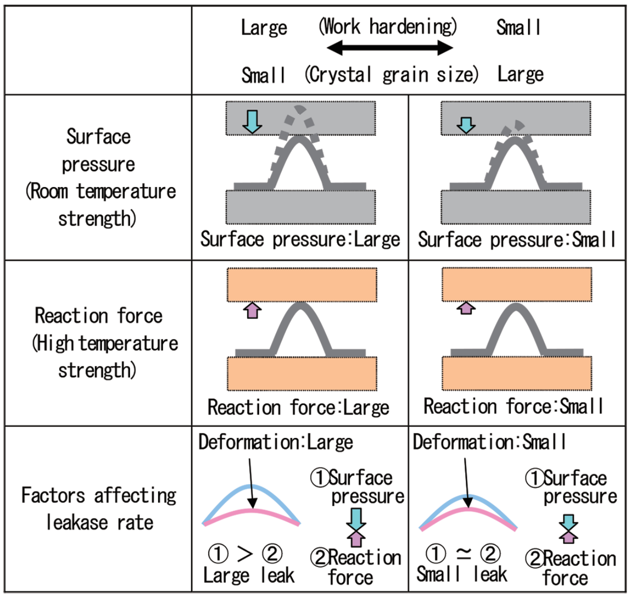

Fig. 13 Schematic of surface pressure and reaction force in 700°C heating evaluation

that the force applied to the materials during bolt tightening differs with the materials. (This force is equivalent to the creep stress during high-temperature heating.) In addition, the force resisting the force applied to the materials differs with the materials. (This force is equivalent to the medium temperature strength and creep strength of the materials.) It was revealed that when the ΔHV (difference between the initial material hardness and the material hardness after heating at 700°C for 120 h) was applied as a simple index, the smaller the ΔHV, the smaller the leak amount became. It is necessary to restrain the depression and surface roughening of the bead top as factors directly affecting the leak resistance.

4. Conclusions

To select the gasket materials that can be applied in the medium-temperature range, we investigated various controlling factors that affect the gasket properties by using the proposed material NSSC 302BN. In addition, we obtained guidelines for selecting gasket materials applicable in the mid-temperature range by focusing on the high-temperature permanent set amount and the bead top shape, two important indexes for understanding the gasket properties.

(1) The residual bead height after heating at 700°C is smaller for materials with high room-temperature strength, high work hardening materials, and ultrafine-grained materials.

(2) The permanent set amount after heating at 700°C and the leak amount are related such that the leak amount is smaller for steels with higher high-temperature leak resistance.

(3) The permanent set and spread of the bead top after heating at 700°C were recognized for steels with lower high-temperature permanent set resistance and finer grain size. The surface roughening of the bead top after shaping was retained after heating.

(4) According to the above study results, we developed the NSSC 302BN. Because the NSSC 302BN proved to have good formability into gaskets and high durability as evaluated by a bench test simulating an actual environment, it is now adopted as material for gaskets for use in the medium-temperature range.

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