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The body can be viewed from the next page.

High Formability R429EX and Heat-Resistant R444EX Stainless Steels for Automotive Exhaust Manifold*


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1 Introduction

Automotive exhaust manifolds fabricated from stainless steel pipe[1,2] such as type 409 (11%Cr-0.2%Ti) and type 430J1L (17%Cr-0.4%Nb-0.5%Cu) have been used in recent years in place of cast iron manifolds. This change corresponds to the rise in exhaust gas temperature resulting from higher engine performance and the decrease in car body weight. This trend is deemed to continue in the future.

However, two major improvements are required of the conventional type 430J1L steel. The first requirement is to improve formability without lowering heat resistance. An exhaust manifold has a complex shape, and severe forming is conducted, so that problems such as cracking during forming and local thickness reduction always occur. Therefore, the application of stainless steel pipe to exhaust manifolds is considered to extend by increasing the forming limit, and an improvement of formability is also being examined[3-5] from the standpoint of method, pipe manufacturing, but further improved formability is desired. The second requirement is an improvement of heat resistance. The exhaust gas temperature has already been increased from the present level of 900°C to over 950°C to examine improvements to the exhaust gas cleaning characteristics and to increase the power output of engines, and the development of materials with higher heat resistance that can meet this requirement is needed. In short, an increase in the gas temperature passing through exhaust manifolds is inevitable, and high-formability ferritic stainless steels for general purpose that enable the application of the present exhaust manifold design to be extended are required.

To develop materials with their formability better than the conventional steel type 430J1L and with their heat resistance better than type 430J1L, an investigation was made into the effects of C, Nb, Mo and Cr on the high-temperature properties of stainless steels and their mechanical properties at room temperature. Based on the study results, a high-formability ferritic stainless steel R429EX for general applications, and a high heat-resistant ferritic stainless steel R444EX for high temperature exhaust gas were development. The following report outlines the developments.

2 Effects of Alloying Elements on the High-Temperature Characteristics and Room-Temperature Mechanical Properties of Ferritic Stainless Steels

2.1 Test Materials and Experimental Method

About 20 vacuum-melted 11–19% Cr ferritic stainless steel ingots, each 30 kg in weight, were cast in the laboratory, with the contents of C, Nb and Mo varied as

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Table 1 Chemical composition ranges of specimens used (mass %)

C	N	Cr	Nb	Mo
0.003～0.03	0.01	11～19	0～1.0	0～2.0

shown in **Table 1** for test purposes. Each ingot was divided into two parts; one for flat rolled specimens through hot rolling, annealing and cold rolling, and then brief annealing at 1 000°C to form 2-mm-thick strip sheets. The following test items (1) to (5) were carried out on these sheets. The other part of the ingots was forged into 35 mm × 35 mm bar specimens, and then annealed at 900°C to 1 000°C for 10 min, and tested to determine the reduction in area in a thermal fatigue test and high-temperature tensile test of round bar specimens.

(1) High-Temperature Tensile Test (JIS G 0567)
(2) Charpy Impact Test (JIS Z 2242)
(3) Identification of Precipitates by X-ray Diffraction
The residues were extracted by potentiostatic electrolysis.
(4) Observation of the Microstructure and Measurement of the High-Temperature Strength
The specimens were heat treated for 200 h in a temperature range from 800 to 950°C in the air.
(5) Resistance to Oxidation
The surfaces of specimens each 2 mm *t* × 20 mm *W* × 30 mm *l* were polished to #320, degreased and heated at 800, 900 and 1 000°C for 200 h. An increase in weight due to oxidations was measured after the heating.
(6) Thermal Fatigue Test
The shape of the specimen used is shown in **Fig. 1**, and the test conditions are shown in **Fig. 2**. A thermal fatigue test on the ferritic stainless steel samples conducted at high temperatures as under the present test conditions results in the pressure of the quartz cell for strain detection, causing deformation in the specimens[6] or the generation of cracks at the point of strain detection during the test. This effect of the strain detection system on specimens was eliminated by adopting the specimen shape shown in Fig. 1, so that the strain detection range was not related to a definite cross-section. For this reason, strains larger than those generated in a specimen with a definite cross-section are concentrated in the middle of the specimen, and an evaluation can be made in terms of a conservative amount of strain. The thermal fatigue test used zero load at temperatures of up to 450°C in the first cycle, and the strain was controlled in the subsequent heating cycles to generate a compressive load at the upper set temperature (800°C) and a tensile load at the lower set temperature (100°C). The rate of temperature rise

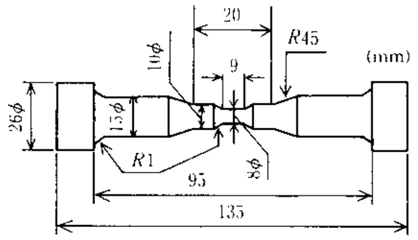


Fig. 1 Dimensions of specimen

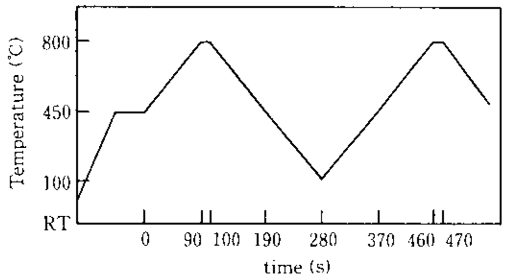


Fig. 2 Condition of thermal fatigue test (1 cycle = 370 s)

was 3.9°C/s, and the holding time at the upper set temperature was 10 s. The number of cycles at the point in time when the maximum tensile load dropped to 70% of the maximum tensile load generated in the fifth cycle was adopted as the point of failure. The total amount of strain applied to the material was calculated by using the following equation, and the gage length for strain detection set at 15 mm:

$$\Delta\varepsilon_t = \Delta\varepsilon_F - \Delta\varepsilon_c \quad \cdots\cdots(1)$$

$\Delta\varepsilon_t$: total strain range
$\Delta\varepsilon_F$: strain equivalent to the amount of free thermal expansion occurring within the maximum and minimum temperature ranges
$\Delta\varepsilon_c$: controlled strain range

2.2 Experimental Results and Discussion

2.2.1 Effects of Nb and Mo on the proof stress at elevated temperatures and on the toughness

The pipe for exhaust manifolds is subjected to such forming operations as bending, expanding and flattening at room temperature. The pipe may sometimes show brittle fracture during forming and, therefore, not only strength at elevated temperatures, but also the low-temperature toughness of the material is important.

Figure 3 shows the effects of the Nb and Mo contents on the high-temperature proof stress at 950°C and on the Charpy impact value at −40°C: for both Nb and Mo, the strength increases with increasing content. Although the absorbed energy decreases with increasing Nb content and Mo content, the degree of decrease in toughness is lower with Mo addition than with Nb addition.

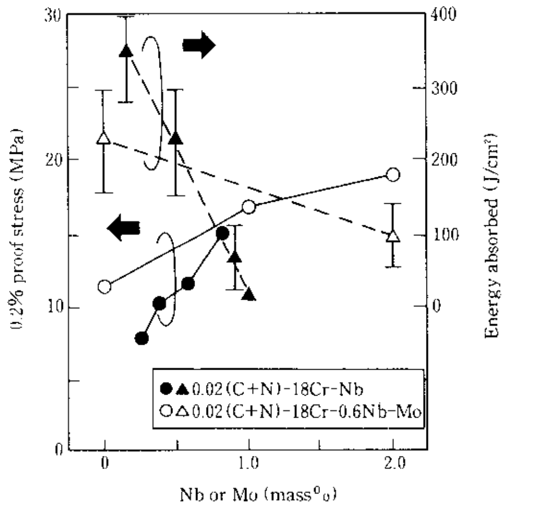


Fig. 3 Effects of Nb and Mo contents on the strength at 950°C and Charpy impact value at −40°C

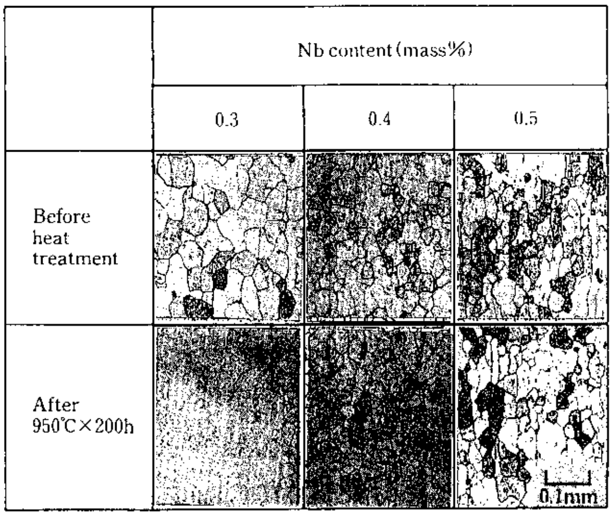


Photo 1 Microstructures of stainless steels before and after heat treatment (0.01 C–0.01 N–18 Cr)

2.2.2 Effect of Cr on the proof stress at elevated temperatures and on the formability

Figure 4 shows the effect of Cr content on the high-temperature proof stress at 900°C, and on the yield strength (YS) and elongation (El) to fracture at room temperature. The Cr content has very little effect on the proof stress at 900°C, while YS at room temperature decreases and El improves with decreasing Cr content, with the result that formability improves.

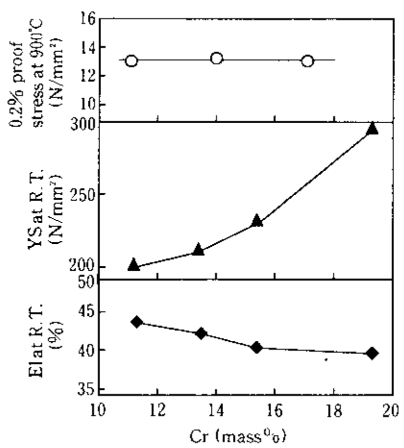


Fig. 4 Effect of Cr content on the 0.2% proof stress at 900°C, YS and El at room temperature (0.2% Ti steels)

2.2.3 Effects of C, Nb and Mo on structural stability at elevated temperatures

To evaluate the structural stability when the steel are used as the material for exhaust manifolds, an investigation was made into the effects of each element on the structural changes after a long-time heat treatment at 950°C for 200 h. **Photo 1** shows the microstructure of longitudinal sections of a cold-rolled annealed 0.01%C–0.01%N–18%Cr steel sheet before and after the long-time heat treatment. The Nb content in this steel was varied at the three levels of 0.3, 0.4 and 0.5%. The grain size is increased greatly by the long-time heat treatment in the 0.3% Nb steel, while this change is small in the 0.4% Nb steel and 0.5% Nb steel. **Photo 2** shows the microstructure of longitudinal sections of the type 430J1L and 18% Cr steel with varied C, Nb and Mo contents before and after the long-time heat treatment. It is apparent that Mo addition and increasing C content do not contribute to structural stability. In contrast, the structural stability at elevated temperatures is found when the C content is reduced and 0.5–0.6% Nb is added.

It is known that suppressing grain growth by fine precipitates is effective for preventing grain coarsening at elevated temperatures. Consequently, in a cold-rolled annealed 18% Cr ferritic stainless steel sheet with the N content fixed at 0.01%, the relationship between the kinds of precipitate and the C and Nb contents was investigated by X-ray diffraction of the electrolytically extracted residues. The detected precipitates are shown in **Fig. 5**. M_6C and Fe_2Nb were detected in the low-C, high-Nb steel. The fact that M_6C and Fe_2Nb are finer than NbC, and that the strength and structural stability at elevated temperatures are both improved by Nb addition can imply that either or both of the M_6C and Fe_2Nb precipitates contribute to these characteristics. However, no Mo precipitates were observed, so that Mo may not be effective in preventing grain coarsening, and the increase in strength at elevated temperatures may be attributable to solute Mo.

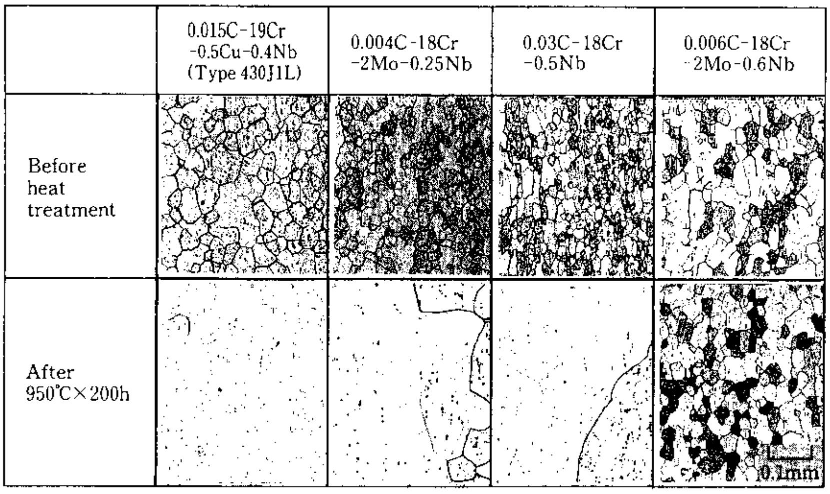


Photo 2 Microstructures of stainless steels before and after heat treatment

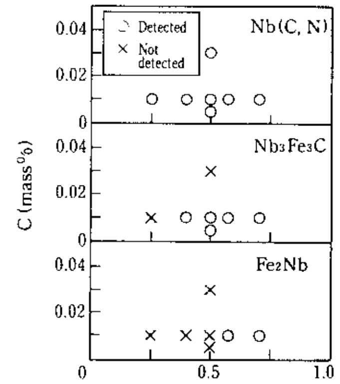


Fig. 5 Identification of precipitates

3 Composition Designing of Developed Steels and their Properties

3.1 Composition Designing of Developed Steels

For the composition designing of high-formability steel for general purpose use, a high-temperature strength equivalent to that of type 430J1L was obtained by not adding expensive Mo and, instead, by adding Nb alone. Furthermore, since the temperature at which an exhaust manifold is used is generally about 900°C at the most, the Cr content was reduced, compared with that in type 430J1L, to a level that still ensured resistance to oxidation at this temperature whereby the formability was improved. The resulting high-formability ferritic stainless steel for general purpose use is RIVER LITE 429EX, the basic composition of which is 15%Cr–0.5%Nb.

It is necessary for a heat-resistant ferritic stainless steel that can withstand high exhaust gas temperatures to have better high-temperature strength than that of type 430J1L. Although the addition of Nb or Mo is effective for improving high-temperature strength,[7-11] the addition of Mo is necessary to improve high-temperature toughness, and the addition of Nb is necessary to ensure structural stability at high temperatures. The resulting steel with resistance to oxidation and ease of manufacture is RIVER LITE 444EX heat-resistant ferritic stainless steel for high exhaust gas temperatures, the basic composition of which is 19%Cr–0.6%Nb–2% Mo, to which both Nb and Mo are added. The chemical compositions of R429EX, R444EX and the conventional steels as the materials for exhaust manifolds are shown in **Table 2**.

Table 2 Chemical compositions of specimens used (mass %)

Specimens	C	Cr	Nb	Ti	Mo	Cu
R429EX[a]	0.010	15.0	0.45	—	—	—
R444EX[a]	0.006	19.2	0.56	—	2.0	—
Type 409	0.011	11.5	—	0.23	—	—
Type 430J1L	0.015	19.1	0.42	—	—	0.54
Type 436	0.010	17.2	—	0.28	1.3	—

[a]Developed steel

3.2 High-Temperature Strength of the Developed Steels

The high-temperature tensile strength and proof stress of various types of ferritic stainless steels are shown in **Fig. 6**. An enlarged figure for the proof stress in the temperature range of 800 to 950°C is shown in **Fig. 7**. R429EX has high-temperature strength equivalent to that of type 430J1L. R444EX has better high-temperature strength than type 430J1L, type 436 and type 409 throughout the whole temperature range, and this suggests the possibility that this steel may be used at higher temperatures than the conventional steels.

The R444EX, R429EX, type 430J1L and type 436 steels were air-cooled after holding for 200 h at 800 to 950°C, and the high-temperature strength of each was measured at 900 and 950°C. The results of the measure-

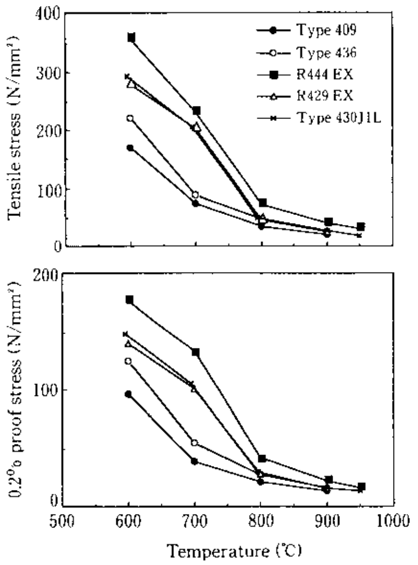


Fig. 6 Tensile and 0.2% proof stress of stainless steels at elevated temperatures

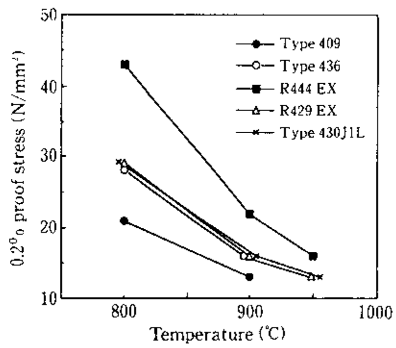


Fig. 7 0.2% proof stress of stainless steels at elevated temperatures

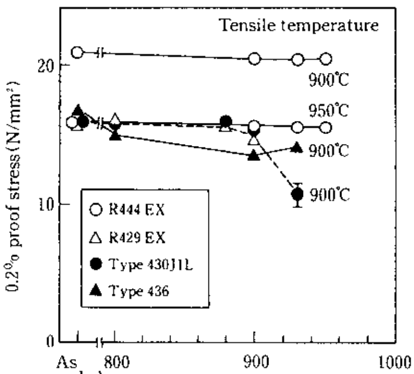


Fig. 8 Effect of reheating temperatures for 200 h on 0.2% proof stress of stainless steels at 900°C and 950°C

ment are shown in **Fig. 8**. R429EX shows softening behavior similar to that of type 430J1L, and the

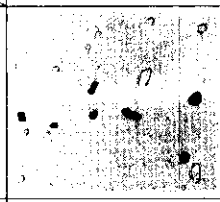
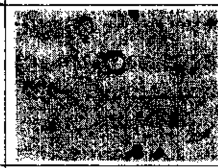
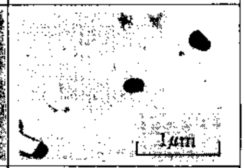
		Before heat treatment	After 930℃×200 h
Type 430J1L	TEM image		
	Insol. Nb	0.19	0.17
		Before heat treatment	After 950℃×200 h
R444 EX	TEM image		
	Insol. Nb	0.26	0.33

Photo 3 TEM images and amount of Nb precipitates before and after heat treatment

decrease in proof stress at 900°C, even after the aging treatment at 900°C for 200 h, is less than 10%. For R444EX, no decrease in proof stress at 900 and 950°C is apparent even after the high-temperature heat treatment, for example, at 950°C for 200 h. Although type 436 shows a slight decrease in proof stress at 900°C after the heat treatment at 800°C or more, the proof stress does not decrease further after heat treatment at higher temperatures.

The results of TEM observations before and after the long-time heat treatment are shown in **Photo 3**. Although no change in the amount of precipitated Nb before and after the heat treatment at 930°C is apparent in type 430J1L, the precipitated Nb after the heat treatment has greatly coagulated and its particles coarsened to a great extent. Such coagulation and coarsening of precipitates are hardly apparent in R444EX that shows no decrease in high-temperature proof stress. The reason why the decrease in proof stress by the long-time heat treatment at 800°C or more is small for type 436, which is a Ti- and Mo-bearing steel, may be that, like R444EX, the high-temperature proof stress that is enhanced by solute Mo is maintained.

The foregoing results infer that the improved high-temperature proof stress at 900°C or more is attributable to fine Nb precipitates, mainly Fe_2Nb-M_6C at ultra-low carbon levels, and to solute Mo when the structural stability is also considered. Furthermore, it seems that the increase in strength at 900°C or more by Mo addition was more stable because of the lower service temperature and smaller time dependence than that by Nb addition.

Therefore, when attempting to increase high-temperature strength by adding only Nb, it may be impossible to maintain high-temperature strength over the long term unless the Nb and C levels are suited to the temperature of the environment in which the steel is used.

3.3 Thermal Fatigue Characteristics of the Developed Steels and the Mechanism for Improvement

Figure 9 shows changes in the maximum tensile load for each cycle during the strain-controlled thermal fatigue tests on R444EX, R429EX and type 430J1L at 100 to 800°C. Under the test conditions, a necking due to plastic deformation was formed in the reduced section of each specimen. **Photo 4** shows fatigue cracks penetrating grains that were observed after the thermal fatigue test on the specimens. During thermal fatigue tests on a ferritic stainless steel at 100 to 800°C, the specimen undergoes a complex process in which the recovery and recrystallization during a temperature rise and the work hardening during a temperature drop are repeated with increasing number of cycles to failure, the sectional area of the specimen is reduced, and fatigue cracks are initiated and propagated to failure.

R444EX has high strength at elevated temperatures, so that loads larger than those applied to R429EX and type 430J1L were applied to this steel in each cycle. The maximum tensile load generated during one cycle depended on the tensile strain generated at the minimum temperature of 100°C.

The relationship between the total strain range and the number of cycles to failure for R444EX, R429EX, type 430J1L and type 409 is shown in **Fig. 10**. Since exhaust manifolds are subjected to thermal strain due to the heating cycle resulting from the starting and stopping the engine, these parts are designed so as to minimize thermal strain. In the total strain range of about 0.3% examined in this experiment, the thermal fatigue characteristic of R429EX and R444EX are better than those of the conventional type 430J1L steel.

In general, the following equation for thermal fatigue characteristics proposed by Manson[12)] and Coffin[13)] applies:

$$\Delta\varepsilon_p \times (N_f)^a = C \quad \cdots\cdots\cdots\cdots(2)$$

a: constant
$\Delta\varepsilon_p$: repreated plastic strain range
N_f: number of cycles to failure
C: material constant

An analysis by Oku et al.[6)] using the above equation has been reported to evaluate the thermal fatigue characteristic of ferritic stainless steels for exhaust manifolds. They explain from Eq. (2) that the thermal fatigue life improves according to this equation because the range of inelastic strain applied to the material (in the plastic strain range) becomes narrow due to an increase in the high-temperature strength of the material. However, the reason for type 409 with low high-temperature strength having the longest life under test conditions with as large a strain as 0.8% of the total strain range is not explained by this.

It is said that, when the high-temperature fatigue characteristics are examined in terms of the total strain range, ductility has a strong effect in the high-strain range, while strength has a strong effect in the low strain range.[14,15)] Furthermore, it is known that C on the right side of Eq. (2) is related to the reduction of area in the tensile test as shown below.[12,13)]

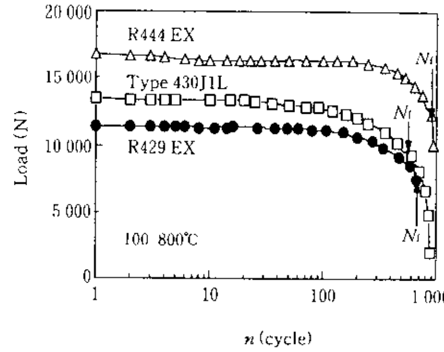


Fig. 9 Change in maximum tensile load with increasing thermal fatigue cycles (N_f: number of cycles to failure)

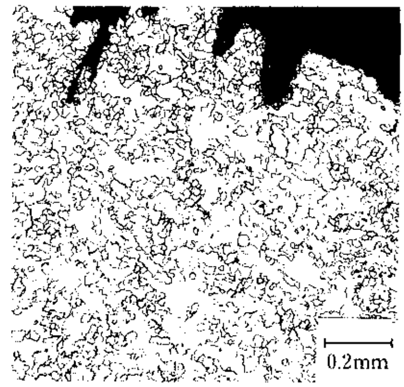


Photo 4 Microstructure of type 430J1L after thermal fatigue test (100–800°C, $\Delta\varepsilon_t = 0.5\%$, $N_f = 550$)

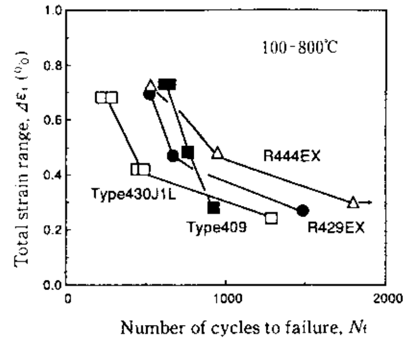


Fig. 10 Relation between total strain range and number of cycles to failure

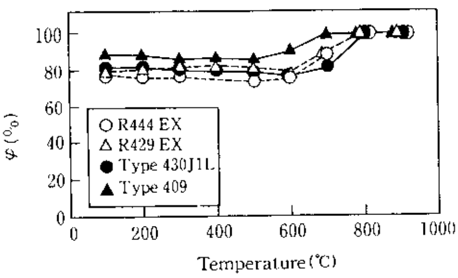


Fig. 11 Reduction of area at elevated temperatures

$$C = \frac{1}{2} \ln \frac{1}{1 - \Phi} \cdots\cdots\cdots\cdots\cdots\cdots (3)$$

Φ: reduction of area to rupture in the tensile test

It is considered that, the larger the reduction of area to rupture in the tensile test, the larger the constant C, with the result that the thermal fatigue life improves. **Figure 11** shows the results of measurements of the reduction of area to rupture by the tensile test on various steel grades. For all the steel grades, the reduction of area reaches a minimum at about 500 to 600°C. Type 409 shows the highest reduction of area throughout the whole temperature range, and the reduction of area decreases in the order of R429EX, type 430J1L and R444EX. It seems that the initiation of cracks during the thermal fatigue test is governed by the balance between an increase in the tensile strain as the temperature drops and the dependence of the reduction in area on temperature. Therefore, this high ductility can partly account for type 409 showing such high fatigue life in the thermal fatigue test in a high-strain range. However, a more detailed examination is necessary to confirm this.

It is apparent from the foregoing discussion that, qualitatively, an increase in strength and improved ductility in the thermal fatigue temperature range would be effective for improving the thermal fatigue characteristics of a material. Furthermore, high-temperature strength and dcutility may sometimes change during long-time heating depending on the heating temperature and time;[11] this makes more complex an evaluation of the thermal fatigue characteristics of ferritic stainless steels for exhaust manifolds. R429EX has high-temperature strength and structural stability equivalent to those of type 430J1L and, at the same time, offers high ductility over a wide temperature range because of the low Cr content. R429EX provides better thermal fatigue characteristics than type 430J1L at 100 to 800°C, irrespective of the strain range. R444EX has higher strength at elevated temperatures than type 430J1L, and provides high ductility close to that of type 430J1L, in spite of the small decrease in high-temperature strength after long-time high-temperature heat treatment. Therefore, the thermal fatigue characteristics of R444EX are about twice better than that of type 430J1L, irrespective of the extent of the strain range. Accordingly, R444EX is a promising material for exhaust manifolds for high exhaust gas temperatures.

3.4 Resistance to Oxidation of the Developed Steels

The results of a 200-h continuous oxidation test at 800, 900 and 1 000°C in the air are shown in **Fig. 12**. R429EX has outstanding resistance to oxidation in the present service temperature range for exhaust manifolds of 750 to 900°C. R444EX provides better resistance to oxidation than type 430J1L and shows outstanding resistance to oxidation in a 200-h continuous oxidation test at 1 000°C.

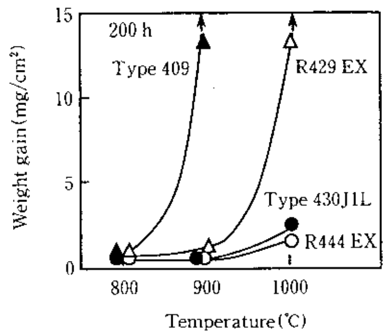


Fig. 12 Effect of temperatures on oxidation properties between 800°C and 1 000°C for 200 h

3.5 Formability of the Developed Steels

Table 3 shows the mechanical properties of R429EX and R444EX at room temperature, and the formability of ERW pipe of these steels, together with data for type 409 and type 430J1L. YS of R429EX is between type 409 and type 430J1L and, therefore, R429EX can be worked more easily than type 430J1L can. The formability of pipe is such that both R429EX and R444EX permit working ranging from flattening to tight forming and can withstand segment pipe expanding up to 1.2 D. Thus, the formability of both developed steels is excel-

Table 3 Mechanical properties of plates and ERW pipes of stainless steels

	Plate (2 mm t)			Pipe (Φ48.6×2 mm t)	
	YS (N/mm²)	TS (N/mm²)	El (%)	1.2D segment expanding	Flattening test
R429EX	299	476	38	Good	2t
R444EX	336	519	36	Good	2t
Type 430J1L	325	474	36	Good	2t
Type 409	224	400	40	Good	2t

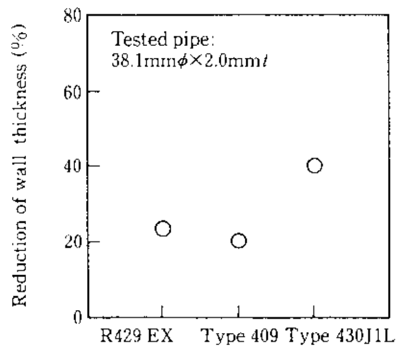


Fig. 13 Reduction of wall-thickness after bending test (40 mm*R* – 90° bending)

lent. **Figure 13** shows the reduction ratios in wall thickness obtained when pipes of R429EX, type 430J1L and type 409 were bent. Even after severe forming, with a reduction in wall thickness of more than 40% for type 430J1L, a reduction in wall thickness of less than 25%, which is almost equal to that for type 409, is maintained with R429EX; this shows that pipe of R429EX has high formability close to that of type 409.

4 Conclusions

To improve the formability and heat resistance of ferritic stainless steels for automotive exhaust manifolds, the effects of the alloying elements C, Cr, Nb and Mo on the high-temperature strength, toughness and high-temperature structural stability were investigated, and the high-formability ferritic stainless steel for general purpose R429EX (15%Cr-0.5%Nb steel) and the heat-resistant ferritic stairless steel for high exhaust gas temperatures R444EX (19%Cr-0.6%Nb-2%Mo steel) were developed based on the result of this investigation. The following knowledge was obtained from the development of these steels:

(1) The addition of Mo and Nb is effective in improving high-temperature strength at temperatures 800°C and above.
(2) Although an increase in the Cr content raises strength, it does not affect high-temperature strength at temperatures 800°C and above.
(3) When high-temperature resistivity at 950°C is to be improved by the same degree by adding Mo or Nb, the deterioration in toughness is smaller with Mo addition than with Nb addition.
(4) Mo addition is not effective for suppressing grain coarsening after the heat treatment at 950°C for 200 h. A decrease in the C content or an increase in the Nb content is both effective in this respect. This seems to be due to the pinning effect of the grain boundaries by such fine precipitates as M_6C and Fe_2Nb.
(5) Improvement in high-temperature resistivity and ductility is both effective for improving the thermal fatigue characteristics.
(6) The high-formability, heat-resistant ferritic stainless steel for general purpose, R429EX, provides high-temperature strength equivalent to that of conventional type 430J1L steel in the form of both sheet and pipe and, at the same time, has high formability superior to that of type 430J1L and close to that of type 409. Furthermore, the thermal fatigue characteristics of R429EX are superior to those of type 430J1L.
(7) After a long-time heat treatment at 950°C, the high-temperature characteristics of R444EX that was developed as a heat-resistant ferritic stainless steel for high exhaust gas temperatures are equivalent to those of type 430J1L after a long-time heat treatment at 900°C. Thus, R444EX shows outstanding high-temperature strength and structural stability after heat treatment at elevated temperatures for a long time. Furthermore, the thermal fatigue characteristics of R444EX are about twice as good as those of type 430J1L. In addition, the formability and resistance to oxidation of R444EX are equivalent to those of type 430J1L.

R429EX expands the scope of application of stainless steel exhaust manifolds as a high-formability material for general purpose use that is suited to more complex shapes than those possible with the conventional steel material. R444EX is suited to higher temperatures and for thinner wall thicknesses of exhaust manifolds which are likely in the future. Both steels are expected to be used in wide applications.

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