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Advanced Stainless Steels for Stricter Regulations of Automotive Exhaust Gas

Atsushi Miyazaki, Junichiro Hirasawa, Susumu Satoh

Synopsis :

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Synopsis:

The demands for advanced properties of stainless steels in automotive exhaust systems are increasing to meet the requirements for lighter weight and stricter regulations for exhaust gas. However, the properties of steel materials used for automotive exhaust systems differ depending on parts, where the materials are to be used. Under the circumstance, advanced stainless steels suitable for each of the parts were developed by Kawasaki Steel by making full use of the latest production facilities in Chiba Works. That is, a stainless steel (R429EX: 15Cr-0.9Si-0.45Nb), having properties excellent in thermal fatigue resistance, resistance to high-temperature fatigue, oxidation resistance and formability, suitable for parts used in high temperature environment, like exhaust manifolds, for example, and stainless steels (R436LT: 18Cr-1.2Mo-Ti and R432LTM: 18Cr-0.5Mo-Ti), having condensate corrosion resistance, appropriate for use in humid environment, like parts, such as, mufflers, have been developed.

1 Introduction

In recent years, with attention focused on global environmental problems, improved automobile fuel economy and exhaust gas purification ratios have been strongly required. In particular, in response to recent stricter emission regulations, improved exhaust gas purification characteristics immediately after the engine starts (cold emissions) have become important. This is because the temperature at the catalytic converter is low and it is therefore difficult to proceed an exhaust gas purification reaction immediately after starting. As a countermeasure for these problems, raising the exhaust gas temperature and adopting thinner materials for the exhaust manifold (for low heat capacity and weight reduction) have been energetically studied. These measures are considered one effective method of improving the exhaust gas purification rate by raising the temperature of the catalyst in the initial period immediately after starting, and can also improve fuel economy by reducing weight. Because the thermal load on the material becomes severe when the exhaust gas temperature increases and the material thickness is reduced in this manner, materials which possess high heat resistance are now particularly required in exhaust manifolds.

In order to satisfy stricter exhaust gas regulations, it has become the mainstream practice to mount a 3-way catalyst in catalytic converters, and as a result, highly corrosive condensates are now generated inside mufflers. Thus, to meet the requirements of longer guarantee periods, materials which offer excellent corrosion resistance in the condensate corrosion environment inside mufflers are also now required.

Under these conditions, Kawasaki Steel developed materials which are suitable for various parts of the automotive exhaust system. Recently developed products include R429EX,[1,2] which offers excellent thermal fatigue resistance, high temperature fatigue resistance, oxidation resistance, and formability, and is suitable for exhaust manifolds, and R436LT and R432LTM,[1,2] which have excellent condensate corrosion resistance for mufflers. The representative chemical compositions of these newly developed steels and conventional steels for automotive exhaust systems are shown in **Table 1**. This report describes the main characteristics of the newly developed steels and the concepts applied in improving properties.

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Table 1 Examples of chemical compositions

(mass%)

Standard designation Kawasaki Steel standard	JIS	C	Si	Mn	Cr	Mo	Ti	Nb
R409L	SUH409L	0.01	0.3	0.3	11.2	—	0.3	—
R429EX		0.01	0.9	0.4	14.8	—	—	0.45
R430LNM	SUS436J1L	0.01	0.3	0.3	17.5	0.5	—	0.38
R436LT	SUS436L	0.01	0.1	0.2	17.8	1.2	0.3	—
R432LTM		0.01	0.1	0.2	17.5	0.5	0.3	—
R434LN2	SUS444	0.01	0.3	0.2	19.0	1.9	—	0.35

2 Stainless Steel for Exhaust Manifolds

2.1 Properties Required in Stainless Steel for Exhaust Manifolds

The exhaust manifold collects the high temperature exhaust gas which is discharged from the engines cylinders and leads the gas to the front pipe. In the following, the service environment and properties required in this part will be described in simple terms.

The exhaust manifold must possess excellent oxidation resistance because it is exposed to high temperature exhaust gas. The manifold is also required to have high temperature fatigue resistance because stress is applied to it by vibration from the engine, road, etc. while it is heated to a high temperature. Moreover, because it is installed in the restricted engine space and is restrained by the surrounding parts, there are cases in which free expansion and contraction during heating and cooling due to engine starts and stops are limited. For this reason, excellent thermal fatigue resistance is also an important property. As will be discussed below, because thermal fatigue resistance is heavily dependent on the degree of restraint (restraint ratio) by the surrounding parts, manifold design is oriented toward reducing the restraint ratio, and in many cases, severe forming is applied to the exhaust manifold so that is can be installed in the limited engine space. Thus, formability is also an important required property.

From the viewpoints described above, stainless steels with heat resistance superior to that of the conventionally used Type 409 (11Cr-Ti) have been developed,[1,3,4] and applied practically. However, because there have been a comparatively large number of reports[5~8] on the oxidation resistance and high temperature fatigue resistance of this type of heat resistant stainless steel, this report will discuss the thermal fatigue resistance of Kawasaki Steel's representative ferritic stainless steels for exhaust system parts (R409L, R430LNM, R429EX, and R434LN2). In addition, the mechanism of improvement in the thermal fatigue resistance of a newly developed steel (R429EX) was also studied.

2.2 Specimen Steels

The chemical compositions of the specimen steels are shown in **Table 2**. As test pieces for the high temperature tensile test, samples were taken from cold rolled and annealed sheets with a thickness of 1.5 mm, manufactured by a commercial process. With the test pieces for the thermal fatigue test, heat treatment was performed after reducing the material from a small, 50 kg ingot to a thickness of 30 mm by forging. Heat treatment was performed at 880°C for 5 min with R409L, at 950°C for 5 min with R429EX and R430LNM, and at 980°C for 5 min with R434LN2 to adjust the grains to a size of approximately No. 7 (in accordance with JIS G 0552).

Table 2 Chemical compositions of specimens

(mass%)

Material	C	Si	Cr	Nb	Ti	Mo
R409L	0.010	0.31	11.0	—	0.25	—
R429EX	0.009	0.85	14.8	0.48	—	—
R430LNM	0.011	0.27	17.5	0.37	—	0.55
R434LN2	0.006	0.25	19.0	0.35	—	1.93

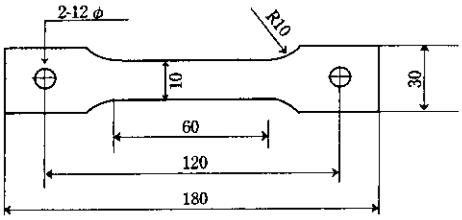
2.3 Evaluation Method

2.3.1 High temperature tensile test

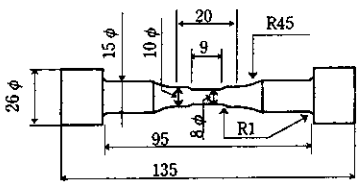
The dimensions of the test piece are shown in **Fig. 1** (a). In conformity with JIS G 0567, the test pieces were held at the specified temperature for 15 min, and the tests were performed at an strain rate of 0.33%/min up to 0.2% strain, and thereafter, at 8.3%/min until failure.

2.3.2 Thermal fatigue resistance

The dimensions of the test piece and the test conditions are shown in Fig. 1 (b) and **Fig. 2**, respectively. Because ferritic stainless steel is comparatively soft at high temperatures such as those applied here, test pieces are susceptible to deformation and cracking during the thermal fatigue test due to the pressure of the quartz rods used to detect strain. Therefore, the surface area of the parts in contact with the strain meter was increased to prevent deformation and breakage from those parts. In the conditions of the thermal fatigue test, as shown in Fig. 2, free thermal expansion was allowed during heating in the initial cycle to an intermediate temperature



(a) Specimen for tensile test at elevated temperatures



(b) Specimen for thermal fatigue test

Fig. 1 Dimensions of specimens

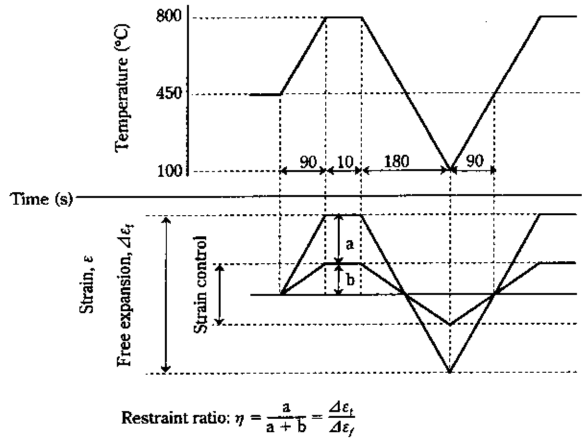


Fig. 2 Condition of thermal fatigue test (1 cycle = 370 s)

between the upper and lower limit temperatures so as not to apply load due to strain control. Then, taking that point as a base line, strain control was introduced. As strain control, the apparent strain ($\Delta\varepsilon_c$) was controlled by an oil hydraulic servo system so as to apply compression at the upper setting temperature and tensile load at the lower setting temperature. In other words, after beginning strain control, thermal expansion and contraction were controlled to a uniform percentage (restraint ratio: η) of free thermal expansion and contraction, as shown in Fig. 2.

$$\eta = \Delta\varepsilon_t/\Delta\varepsilon_f \cdots\cdots (1)$$

$$\Delta\varepsilon_t = \Delta\varepsilon_f - \Delta\varepsilon_c \cdots\cdots (2)$$

Here,

$\Delta\varepsilon_f$: Amount of strain due to free thermal expansion and contraction, measured by the same test apparatus in advance of the test

$\Delta\varepsilon_c$: Amount of apparent strain controlled between quartz rods

The rate of heating was approximately 4°C/s, and the holding time at the upper setting temperature was 10 s. The number of cycles to failure in this experiment was the number of cycles at the point in time when the maximum tensile load had decreased to 70% of the maximum tensile load generated in the 5th cycle, in which the load-strain hysteresis loop had stabilized. The amount of plastic strain was obtained from the hysteresis curve during the 5th cycle. The distance between reference points (gauge length) for strain detection was 15 mm.

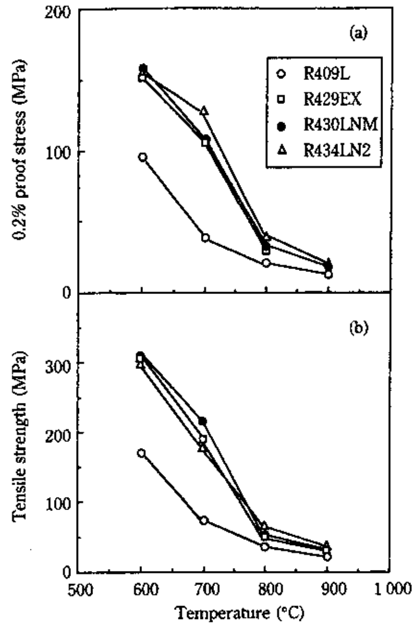


Fig. 3 Strength at elevated temperatures: (a) 0.2% proof stress, (b) Tensile strength

2.4 High Temperature Strength and Thermal Fatigue Resistance

2.4.1 High temperature strength

Figure 3 shows the 0.2% proof stress and tensile stress at elevated temperatures between 600°C and 900°C measured in the high temperature tensile test. At 600°C, in increasing order, the strength of the materials was R409 < R429EX ≒ R434LN2 ≒ R430LNM. At 800°C, the order was R409 < R429EX ≒ R434LN2 < R430LNM.

2.4.2 Thermal fatigue resistance

(1) Relationship between Number of Cycles to Failure and Restraint Ratio

Figure 4 shows the relationship between the num-

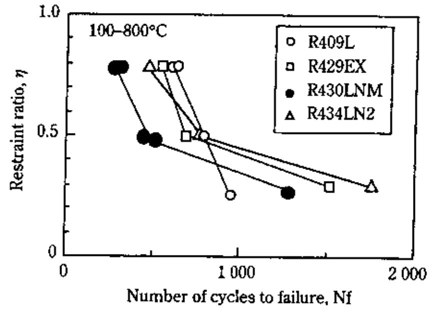


Fig. 4 Relation between restraint ratio and number of cycles to failure in thermal fatigue test

ber of cycles to failure in the thermal fatigue test and the restraint ratio at temperatures between 100°C and 800°C.[9] In the case of high restraint ratios such as 0.8, material life increased in the order R430LNM < R434LN2 < R429EX < R409L. On the other hand, when the restraint ratio was reduced to 0.3, the life of R429EX, R430LNM and R434LN2 improved remarkably, but there was only slight improvement in the life of R409L. At this low restraint ratio, the life of these steels, in increasing order, was R409L < R430LNM, R429EX < R434LN2. Thus, because the relative superiority and inferiority of the respective materials varies depending on the restraint ratio, care is considered necessary in evaluating material life.

(2) Influential Factor on Thermal Fatigue Resistance

The factors which have a large influence on the number of cycles to failure in thermal fatigue tests are generally thought to be strength and elongation.[10–12] Forest et al.[13] have proposed the concept of a plastic strain ratio ($h = \Delta\varepsilon_p/\Delta\varepsilon_t$). In cases when this value exceeds 0.5, elongation is rate controlling for life, whereas, at values under 0.5, strength becomes rate controlling. Because the relative superiority of thermal fatigue life changed depending on the restraint ratio in the measurements made in the thermal fatigue test in the present work, as shown in Fig. 4, it is considered possible that the main factor which determined life varied depending on the restraint ratio. Therefore, a comparison of the plastic strain ratio was made for R409L and R430LNM, which showed a particularly clear reversal in the number of cycles to failure. **Figure 5** shows the relationship between the restraint ratio and the plastic strain ratio (h). At plastic strain ratios of 0.5 and over, it is considered that elongation is rate controlling for life. However, when the restraint ratio was reduced to 0.3, the plastic strain ratio of R409L decreased to approximately 0.6, and that of R430LNM decreased to about 0.3. Thus, with both steels, the plastic stain ratio decreased as the restraint ratio decreased. This is considered to mean that the rate controlling factor for life changes form elongation to strength as the restraint ratio decreases. Therefore, the effect of elongation and strength on the

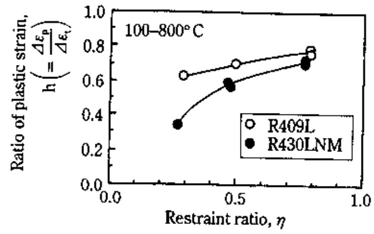


Fig. 5 Relation between ratio of plastic strain and restraint ratio in thermal fatigue test

number of cycles to failure in the thermal fatigue test was studied.

(3) Relationship between Number of Cycles to Failure and Elongation

Generally, thermal fatigue resistance is arranged on the two logarithmic axes of the amount of plastic strain and number of cycles to failure. This is because there are many cases in which the relationship between the two satisfies Eq. (3), which was proposed by Manson[10] and Coffin.[11]

$$N = (C/\Delta\varepsilon_p)^{1/\alpha} \quad \cdots (3)$$

$$C = (1/2)\ln\{1/(1-\phi)\} \quad \cdots (4)$$

ϕ: Reduction of area

The life of materials is decided by the plastic strain range ($\Delta\varepsilon_p$), the term representing elongation (C), and α.

Figure 6 shows the relationship the $\Delta\varepsilon_p$ of R409L and R430LNM, which were measured in the present work, and the number of cycles to failure in the thermal fatigue test plotted on logarithmic axes. It can be understood that a virtually parallel linear relationship was obtained, satisfying Eq. (1). Therefore, the values of α and C for the two steel were obtained from these straight lines, with the results shown in **Table 3**. It is known that the term for elongation, C, in Eq. (3) also depends on the reduction of area of the base material.[10,11] Reduction of area varies greatly depending on temperature, but because the samples received the largest tensile strain at the lower limit temperature in the thermal cycle, it is considered that the characteristic of reduction of area at this time had a large influence on the number of cycles to failure. Therefore, the reduction of area at the lower limit temperature (100°C) was measured in a high temperature tensile test using thermal fatigue test specimens. Table 3 shows a comparison of the reduction of area in this test and the term for elongation, C, which was estimated from the test values, and elongation, C, obtained in the thermal fatigue test. The elongation, C, of R409L obtained from the reduction of area at 100°C was approximately 1.5 times larger than that of R430LNM. The elongation, C, obtained in the high temperature tensile test of R409L and R430LNM

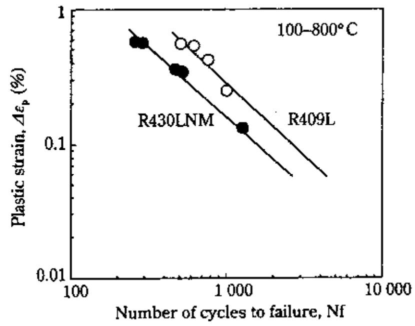


Fig. 6 Relation between plastic strain and number of cycles to failure in thermal fatigue test

Table 3 Comparison between parameters of Manson-Coffin relation and results of tensile test

Material	Resultes of thermal fatigue test $\Delta\varepsilon_p N^{\alpha} = C$		Results of tensile test at 100°C	
	α	C	ϕ	$C = 1/2 \ln 1/(1-\phi)$
R409L	1.0	1.6	0.9	1.2
R430LNM	0.9	0.9	0.8	0.8

showed good agreement with the elongation, C, obtained in the thermal fatigue test, indicating the appropriateness of the argument presented above. On the other hand, α showed approximately the same value with both steels, and the elongation, C, of R409L was approximately 1.8 times larger than that of R430LNM. Therefore, because the plastic strain range, $\Delta\varepsilon_p$, does not differ greatly in the two steels, as shown in Fig. 5, R409L, which has a larger elongation term, C, according to Eq. (3), is considered to be more effective than R430LNM in securing thermal fatigue resistance under conditions in which elongation is rate controlling, namely, under conditions characterized by a high restraint ratio.

(4) Relationship between Number of Cycles to Failure and Strength

As shown in Fig. 5, the plastic strain ratio decreases to 0.5 or under when the restraint ratio becomes low, and the factor which is rate controlling for the number of cycles to failure shifts mainly to strength. In particular, as the plastic strain ratio approaches 0, in other words, as the plastic strain range, $\Delta\varepsilon_p$, approaches 0, a remarkable improvement in the number of cycles to failure, in accordance with $(1/\Delta\varepsilon_p)^{1/\alpha}$, can be expected from Eq. (3). Further, it is known that the parameter $\Delta\varepsilon_p$ in Eq. (3) is influenced by strength.[14,15] With both steels R409L and R430LNM, the plastic strain ratio (plastic strain range) decreases as the restraint ratio decreases. In this process, it is considered that the decrease behavior of the plastic strain ratio in these two steels differs due to differences in the strengths of the steels. Specifically, with regard to the decrease behavior of the plastic strain ratio, R430LNM, which is high in strength in comparison with R409L, shows a remarkable increase in the number of cycles to failure, in accordance with Eq. (1), when the restraint ratio decreased to 0.5 or under because it is reduced from a higher restraint ratio, as shown in Fig. 5, and as a result, there was a reversal in the relative thermal fatigue life of these two steels, as shown in Fig. 4.

2.5 Comparison of Thermal Fatigue Resistance of R429EX and R430LNM

In section 2.4 the thermal fatigue life (number of cycles to failure) of R409L and R430LNM were compared and studied, showing that elongation has a large influence on thermal fatigue life under conditions characterized by high restraint ratios, whereas strength has a large influence at low restraint ratios. Further, under all the restraint ratio conditions which were studied here, the newly developed steel R429EX showed a thermal fatigue life superior even to that R430LNM, which is an 18Cr type stainless steel (Fig. 4). The reason for this is considered to be as follows: Both steels are Nb-added ferritic stainless steels, and in addition to the fact α in Eq. (3) is approximately the same in both, their strength is also on approximately the same level. Thus, the plastic strain range, $\Delta\varepsilon_p$, is also similar. On the other hand, because R429EX is a lower alloy steel, it possesses higher elongation,[1] and the term representing elongation, C, is larger. Therefore, in accordance with Eq. (3), it is considered that the thermal fatigue resistance of R429EX is advantageous.

2.6 Application to Automotive Exhaust System Parts with Formability and Thermal Fatigue Resistance

Because parts of the automotive exhaust system which are used in high temperature environments, as represented by the exhaust manifold, are designed on the precondition of a low restraint ratio relative to the heat cycle,[16] R429EX, R430LNM, and R434LN2 can be expected to show thermal fatigue resistance superior to that of R409L. However, formability deteriorates to the extent that high heat resistance is indicated and higher alloy material compositions are adopted. For this reason, there are also many cases in which it is not possible to form the specified shape, namely, a low restraint ratio shape. Thus, it goes without saying that excellent formability is also important from the viewpoint of improving thermal fatigue resistance. In particular, in recent years, the temperature of the material of the external cover of the catalytic converter has tended to increase with higher exhaust gas temperatures. For this reason, there has been an increasing need for a material with heat resistance superior to that of R409L. Moreover, a high r-value is necessary in many cases because the external cover is

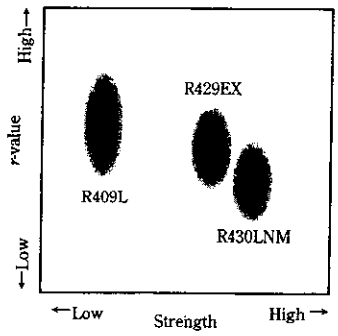


Fig. 7 r-value and strength at room temperature of stainless steels for exhaust manifold

normally manufactured by press forming.

Considering this background, Kawasaki Steel adopted a chemical composition and process that particularly improve the r-value by applying advanced steelmaking technology and hot rolling technology to R429EX, which has the distinctive feature of a relatively low Cr composition. **Figure 7** shows a conceptual diagram of the room temperature r-value and strength of Kawasaki Steel's materials for auto exhaust systems. With R429EX, formability relatively close to that of the conventional steel, R409L, can be expected.

To summarize the above discussion, as a philosophy for the selection of stainless steels for use in high temperature environments such as exhaust manifolds and the external covers of catalytic converters, if attention is given to thermal fatigue resistance and high temperature fatigue resistance,[8] it is possible to select R429EX, R430LNM, or R434LN2, in which high strength is realized by adding Nb or Mo, in cases when the properties of R409L are inadequate. However, R429EX is particularly recommended from the viewpoint of formability and is increasingly adopted for this reason. On the other hand, R430LNM is suitable if priority is given to oxidation resistance, and R434LN2, in which even higher strength is realized by 2% Mo addition, is recommended when excellent thermal fatigue resistance and high temperature fatigue resistance are also necessary.

3 Newly Developed Stainless Steels for Mufflers

In addition to formability and weldability, high corrosion resistance is also required in muffler materials in consideration of the problems of noise and exhaust gas leaks if the muffler is perforated due to corrosion.

Particularly with regard to corrosion of the muffler interior, after the 3-way converter was adopted for use in exhaust gas purification systems, a highly corrosive condensate (condensed exhaust gas) containing NH_4^+ and Cl^- formed inside of the mufflers, and as a result, perforation frequently occurred in Al plated steel. This problem led to the adoption of SUH409L (11%Cr-0.3%Ti) and SUS410L (13%Cr). However, in recent years, with longer guarantee periods for automotive parts, steels with an even higher level of corrosion resistance have become necessary.

We carried out research to elucidate the mechanism of condensate corrosion, and clarified the fact that this type of corrosion is promoted by the following four factors.[17]

(1) The concentration of Cl^- increases and pH is reduced by evaporation and concentration of the condensate, and the corrosiveness of the condensate becomes extremely high.[2]
(2) Corrosion resistance is deteriorated by oxidation.
(3) The soot which is contained in the exhaust gas promotes a cathodic reaction.
(4) In the case of Al plated steels, the galvanic protection effect of Al is blocked by an alloy layer at the interface between the substrate steel and the oxidized layer at the Al plating surface.

Because condensate corrosion of this type cannot be simulated by the conventional cyclic corrosion test, in which the test material is sprayed with salt water, dried, and then held at high humidity, a test method which simulates condensate corrosion was studied, and the Dip & Dry test method shown in **Fig. 8** and **Table 4** was developed.[18] This test is used to evaluate corrosion resistance by simulating corrosion resulting from the process of concentration and evaporation of the condensate by immersing a test piece, which has been given preparatory heat treatment, in a synthetic condensate, which is prepared based on the analysis of the condensate from actual automobiles. The test piece is then held in the synthetic solution at 80°C, which is the temperature of the muffler when the condensate is present.

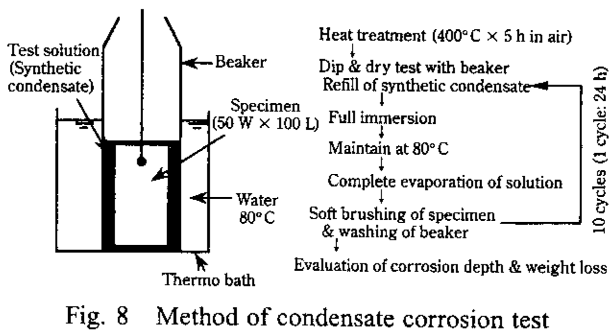


Fig. 8 Method of condensate corrosion test

Table 4 Chemical composition of synthetic condensate

(ppm)

Cl^-	SO_3^{2-}	SO_4^{2-}	CO_3^{2-}	NO_2^-	NO_3^-	CH_3COO^-	HCHO	$COOH^-$	NH_4^+	Activated carbon
250	1 250	1 250	2 000	100	20	400	250	100	2 500	50 g/ℓ

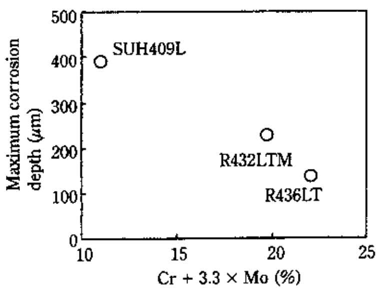


Fig. 9 Effect of Cr and Mo contents on the maximum corrosion depth in the synthetic condensate corrosion test

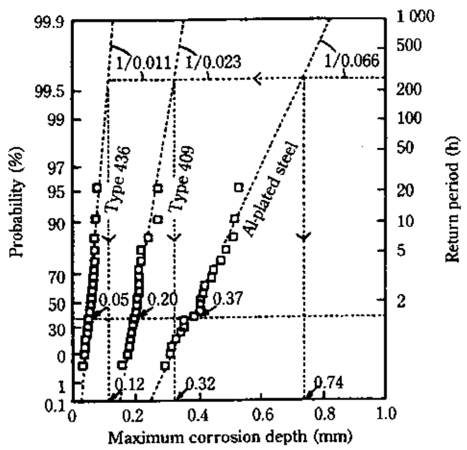


Fig. 10 Doubly exponential probability plots of the maximum corrosion depth of the steels after 10 cycles of condensate corrosion test

Figure 9 shows the results of a simulated condensate corrosion test of stainless steels for mufflers. It can be understood that the maximum corrosion depth, which is linked to perforation in mufflers, decreased in proportion to Cr (%) + 3.3 × Mo (%), and that Cr and Mo are effective in improving condensate corrosion resistance. Because the measured values of the maximum corrosion depth deviate relatively easily, it is dangerous to make a simple estimation of material life to perforation from this value. Therefore, the life of the materials was estimated using a method of statistical analysis of the extreme values in the maximum corrosion depth data. **Figure 10** shows the result of applying a Gumbel probability plot to the maximum corrosion depth data of the stainless steels for muffler use. It can be understood that satisfactory linear relationships were obtained with all the data, and the results were arranged by double exponential distributions. After correction for differences in the surface area with the test pieces, the estimated values of the maximum corrosion depth when this test was applied to actual mufflers were 0.74 mm for Al plated steel, 0.32 mm for SUH409L, and 0.12 mm for R436LT. This test was also conducted by varying the number of

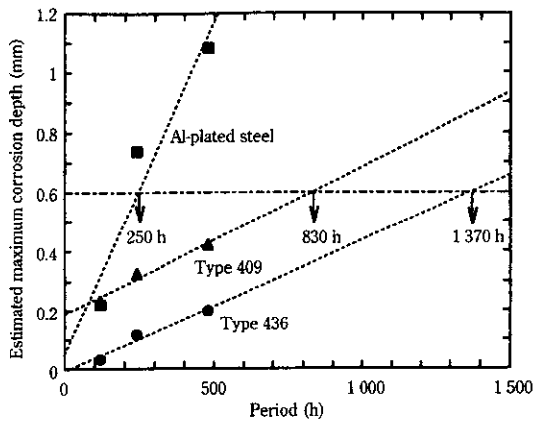


Fig. 11 Relation between estimated maximum corrosion depth and period of the condensate corrosion test

test cycles, and the changes in the estimated maximum corrosion depth which occurred in mufflers as a result of the test time were plotted as shown in **Fig. 11**. The time to complete perforation of the thinnest shell (0.6 mm thickness) which is normally used in mufflers was 250 h with Al plated steel, 830 h with SUH409L, and 1 370 h with R436LT. Thus, it was estimated that the life of SUH409L is 3.3 times longer than that of Al plated steel, and the life of R436LT (18%Cr-1.2%Mo-0.3Ti) is 1.7 times that of SUH409L.

Based on these results, R436LT and R432LTM (18%Cr-0.5%Mo-0.3%Ti), in which Mo is added to 18%Cr steel, were developed as high corrosion resistance muffler materials. These steels are manufactured using high productivity, low cost production equipment for carbon steel (tandem mill cold rolling, with annealing and pickling on a continuous annealing line for carbon steel), and are being widely used as muffler materials.

4 Conclusion

As a result of the stricter regulations applied to automobile exhaust gas in recent years, the service environment for stainless steels have become increasingly severe. Newly developed steels include R429EX (15Cr-0.9Si-0.45Nb), which offers excellent thermal fatigue resistance, high temperature fatigue resistance, oxidation resistance, and formability, and is used as a material for high temperature environments represented by the exhaust manifold, and R436LT (18Cr-1.2Mo-Ti) and R432LTM (18Cr-0.5Mo-Ti), which provide excellent condensate corrosion resistance and are used in humid environments represented by the muffler. In particular, this report has described the representative properties and mechanisms of improvement in the properties of these newly developed steels.

In the future, a trend toward stricter exhaust gas regulations is foreseen in all countries. It is expected that high performance stainless steels which are suited to these stricter regulations will be used even more widely in the future and will contribute to a cleaner environment.

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