Study on Dispersion Strengthened Ferritic Heat Resisting Steel*

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Synopsis

The sintering characteristics and mechanical properties of the ferritic heat-resisting steel strengthened with finely dispersed phase of aluminium oxide were investigated and the following results were obtained.

(1) A small amount of addition of ferro-titanium alloy powder, which yields liquid phase at the sintering temperature, is effective for the densification of the sintered material and for improving the distribution of aluminium oxide particles.

(2) Hot working of the sintered materials is effective for the improvement of their mechanical properties, especially creep rupture strength.

(3) Sintered ferritic heat resisting steels having higher creep rupture strength than that of lower grade austenitic steels are obtained with a combination of the above-mentioned production methods.

I. Introduction

Dispersion strengthened alloy with very fine particles of non-metallic substances such as oxides and carbides artificially dispersed in the metallic matrix has been attracting much attention as a new type of heat resisting material. An outstanding feature of this type of alloy is that it maintains superior strength over a long period at elevated temperatures, since neither the coagulation of the dispersed particles nor the solution of those particles into the matrix occur, as was found in the sintered aluminium products (SAP) developed by Irmann.¹⁾

For this reason, the investigation into this new alloy has been conducted by many researchers for the purpose of developing new material or making clear the strengthening mechanism theoretically and various reports have been published up to date.²⁾⁻⁷⁾ It was also reported that TD Nickel*** which is a two phase alloy containing approximately 2 vol% thoria (ThO₂) dispersed in a matrix of pure nickel has been practically used for the construction of after-burner of the jet engine and gas turbine nozzle guide vanes.⁸⁾ As this type of alloys, however, is still at its early stage of development as a whole, the application of dispersion strengthening method has been mostly attempted at pure metals and seldom at alloy steels. Consequently the utilization of this type of material is limited to some special purposes.

On the other hand, the costly austenitic heat resist-

ing steels are still generally employed for the service under high stress at elevated temperature.

However, if the ferritic materials can be strengthened as comparable to the austenitic steel, it will no doubt bring about a large improvement in the field of heat resisting material both technically and economically.

The purpose of the present work is to develop the dispersion strengthened ferritic steel having the elevated temperature strength well comparable to that of the austenitic heat resisting steels. As the first step, the authors investigated the sintering characteristics of 13%Cr steels with finely dispersed aluminium oxide particles and then tried to improve the mechanical properties, especially the elevated temperature strength by employing the technique of the liquid phase sintering and hot-working the sintered compact.

II. Experimental Procedure

For the improvement of the dispersion strengthened alloys, various techniques have hitherto been proposed by many researchers. In this study, the mechanical mixing method, the most usual one among various techniques, was employed. As powder to form the matrix, prealloyed 13CrMoTi and AISI 406 steel powders (—150 mesh) having chemical compositions as shown in Table 1 were used. The reason for using these two kinds of powders was to compare the effect of matrix on the dispersion strengthening. As the characteristics of the powder for the dispersion particles have of course a large effect on the mechanical properties of the sintered compact, a very fine particle of γ -Al₂O₃ with average grain size of 20 m μ was selected as one of the most proper kind of dispersion particles. Table 2 shows the physical and chemical properties of γ -Al₂O₃ used.

Photographs 1 and 2 show the electron microstructure and electron diffraction pattern of γ -Al₂O₃, respectively. The observed lattice spacings of γ -Al₂O₃, together with the ASTM data, are shown in Table 3.

In addition, Fe-Ti alloy powder (26.61 wt% Ti, —325 mesh) which yields liquid phase at the proper sintering temperature of the steel powders selected was

Table 1. Chemical composition of alloy steel powders

Powder	C	Si	Mn	S	P	Cr	Al	Mo	Ti
AISI 406 steel	0.03	0.64	0.69	0.017	0.013	12.91	2.86	—	—
13CrMoTi steel	0.04	1.35	0.15	0.02	0.022	12.91	0.14	1.91	0.41

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used, in order to improve the sintering characteristics.
Mixing of powders was carried out in horizontal ball

Photo. 1. Electron photomicrograph of γ - Al_2O_3 used ($\times 5\,000$) (5/6)

Photo. 2. Electron diffraction pattern of γ - Al_2O_3

Table 2. Physical and chemical properties of γ - Al_2O_3

Surface area	100 $\pm$ 10 m ² /g (BET method)
Grain size	5 to 30 m μ
Specific gravity	3.4
Purity	Al ₂ O ₃ 99% min. HCl 1% max. Fe ₂ O ₃ 0.05 max. SiO ₂ } Not detected TiO ₂ }

Table 3. Observed lattice spacings

Observed d (Å)		ASTM data on γ - Al_2O_3 d (Å)	
d (Å)	Intensity	d (Å)	I/I_0
2.78	<i>W</i>	2.80	20
2.39	<i>m</i>	2.39	80
2.30	<i>W</i>	2.28	50
1.99	<i>S</i>	1.977	100
1.52	<i>VW</i>	1.520	30
1.40	<i>VS</i>	1.395	100
1.14	<i>VW</i>	1.140	20
1.06	<i>VW</i>	1.027	10
0.877	<i>VW</i>	0.884	10
0.804	<i>VW</i>	0.806	20

Note: *VW*: very weak *W*: weak *m*: medium
S: strong *VS*: very strong

mill by the wet process using carbon tetrachloride. The mixtures were dried at about 100°C and then annealed for one hour at 800°C in the purified hydrogen to improve the compressibility. As a lubricant, 0.5 to 1.0% of zinc stearate or liquid paraffin was added to the annealed mixtures, prior to compacting them. In compacting the powders, either a die with a rectangle-shaped hole, 18 mm in width and 100 mm in length, or a die having a cylindrical hole, 45 mm in diameter was used, depending on the experimental purpose. Compacting pressure was 8 t/cm² and the height of green compact to be obtained was adjusted by the weight of powders.

A preliminary test was made on the green compact to find the optimum sintering condition for each material employed. As a result, it was decided as shown in Table 4.

Moreover, the effect of hot working on the mechanical properties of the sintered compacts was investigated by forging them, either in the state as sintered or sintered and sheathed in a mild steel case.*

III. Experimental Results

1. Effect of Sintering Condition on the Mechanical Properties of Sintered Compacts

As the results of the preliminary test, Fig. 1 shows

Table 4. Sintering condition

Matrix	Sintering temperature	Sintering time	Sintering atmosphere
AISI 406 steel	1 400°C	5 hr	Purified hydrogen
13CrMoTi steel	1 350°C	5 hr	Vacuum*

* Degree of vacuum: (2-5) $\times 10^{-4}$ Torr. at sintering temperature

Fig. 1. Effect of sintering temperature and sintering atmosphere on mechanical properties of sintered 13CrMoTi steels

* The latter will be referred to as sheath forging to be discriminated from the former.

the effect of the sintering temperatures and atmospheres on the mechanical properties of the sintered 13CrMoTi steels. In this experiment, the green compacts were sintered for 5 hr at each temperature. The results show that the mechanical properties were gradually improved as sintering temperature was raised. Regarding the effect of sintering atmosphere, the mechanical properties were much more improved by vacuum sintering than sintering in hydrogen atmosphere. Particularly, a remarkable difference can be seen in the tensile elongation. This is due to the higher density and finer grain size in the sintered compact obtained by vacuum sintering.

Sintering was also conducted for 10 hr at 1300° and 1350°C, respectively, for comparing the strengths with those of the compacts sintered for 5 hr. Further, experiments were carried out at or about 1400°C for the purpose of shortening the sintering time, but with no satisfactory results.

Next, similar investigation was also carried out on AISI 406 steel powder compacts and it was learned that holding for 5 hr at 1400°C would be the optimum sintering condition for that steel. Moreover, it was found that the sintering atmosphere gave little effect on the mechanical properties of the sintered AISI 406 steels. The tensile strength and elongation of the sintered material obtained by sintering in hydrogen atmosphere and in vacuum under the above mentioned condition were 45 to 46 kg/mm² and 9 to 10%, respectively for both cases. Compared with the mechanical properties of the sintered 13CrMoTi steel, they are considerably lower in elongation but only slightly lower in tensile strength.

2. Effect of Addition of γ -Al₂O₃ on Mechanical Properties of Sintered Compact

Figure 2 shows the effect of addition of γ -Al₂O₃, with various mixing times in the range of 3 to 72 hr, on the room temperature tensile properties of the sintered 13CrMoTi steel. In this figure, it is seen that the strength of the sintered 13CrMoTi steels with dispersed 0.5% of γ -Al₂O₃ is higher than that of the sintered compacts without addition of γ -Al₂O₃. This fact gives an evidence of the strengthening effect of dispersed γ -Al₂O₃. Regarding the effect of mixing time, the strength of the sintered compacts gradually increased as mixing time was prolonged. Tensile

elongation, on the contrary, showed a slight decrease with the increase of mixing time.

Photograph 3 shows the effect of mixing time on the microstructure of the sintered 13CrMoTi steels with dispersed 0.5% γ -Al₂O₃. Coagulation of γ -Al₂O₃ was remarkable when mixing time was shorter than 15 hr. The trend was moderated as mixing time was prolonged and γ -Al₂O₃ particles were uniformly distributed when the mixing time was increased to 72 hr.

Figure 3 shows the changes of the density and the tensile properties of the sintered AISI 406 steels as a

Fig. 2. Effect of mixing time of γ -Al₂O₃ particles on tensile properties of sintered 13CrMoTi steels

Fig. 3. Relation between amount of γ -Al₂O₃ and sintered density and tensile properties of sintered AISI 406 steels

Photo. 3. Effect of mixing time on microstructure of sintered 13CrMoTi steel with dispersed γ -Al₂O₃ particles ($\times 100$) (5/6)

(a) 3 hr mixing (b) 15 hr mixing (c) 30 hr mixing (d) 72 hr mixing

function of the amount of dispersed $\gamma\text{-Al}_2\text{O}_3$.

It can be seen from this figure that the strengthening effect appears even when only 0.1% of $\gamma\text{-Al}_2\text{O}_3$ was added and there was no remarkable change in the tensile properties of the sintered compact with further increasing of the amount of $\gamma\text{-Al}_2\text{O}_3$ up to 0.5%.

As $\gamma\text{-Al}_2\text{O}_3$ particles used in this experiment were very fine, it was thought that the addition of only 0.5% could form the dispersed phase of a sufficient quantity. However, the authors investigated the strengthening effect in the case of further addition of $\gamma\text{-Al}_2\text{O}_3$ in the sintered 13CrMoTi steel.

Table 5 shows that the tensile strength and elongation of the sintered compact decreased slightly when the amount of $\gamma\text{-Al}_2\text{O}_3$ was increased from 0.5 to 1%. With regard to the mechanical properties of the sintered AISI 406 steel, the same tendency was also found. The strength of the sintered compacts decreased further if $\gamma\text{-Al}_2\text{O}_3$ was added over 1%. Thus, the plan to improve the room temperature strength of the sintered compact by adding a large amount of $\gamma\text{-Al}_2\text{O}_3$ was not successful. In spite of this fact, it was still necessary to increase the amount of $\gamma\text{-Al}_2\text{O}_3$ for improvement of the elevated temperature strength, particularly creep rupture strength, of the sintered compact, as described in the following.

3. Study for Improvement of Dispersion Strengthening Method

Improvement of Sintering Characteristics by Liquid Phase Sintering

It has been well-known that the liquid phase sintering remarkably increases the sintering rate and, as a result, it has an outstanding effect for improving the physical and mechanical properties of the sintered products. The application of this liquid phase sintering to the iron powder products hitherto has been made usually by adding small quantities of boron or phosphorus to the iron powder in the form of powdered

ferro-boron or ferro-phosphorus, each of which yields a liquid phase at the sintering temperature of iron powder.^{9),10)} However, boron has little solubility for α -iron and addition of only 0.1% of it causes hard eutectic constituent to remain at the grain boundaries and as a result, to reduce the toughness of the products. It is, therefore, impossible to increase the addition of boron adequate to make a satisfactory improvement of the sintering characteristics. For the same reason, it is difficult to increase the amount of phosphorus to more than 0.8%.

Considering the high solubility of titanium in α -iron, the authors attempted to improve the sintering characteristics of the concerned alloy steels with or without dispersed $\gamma\text{-Al}_2\text{O}_3$, by a large amount of addition of Fe-Ti alloy which produces the liquid phase at the sintering temperature. Figure 4 shows the effect of addition of Fe-Ti alloy on the tensile properties of 13CrMoTi steel without dispersed $\gamma\text{-Al}_2\text{O}_3$ particle.* It is clear from this figure that addition of Fe-Ti alloy has a remarkable effect to improve the strength of the sintered compact and more than 1% titanium should be added to achieve a sufficient increase in strength. However, addition of more than 2% of titanium causes a slight decrease in the strength.

On the other hand, the tensile elongation was improved with increased addition of titanium up to 0.5%, but it decreased with further addition of it. Nevertheless, the elongation over 10% could be obtained even when titanium addition was amounted to 3%. The effect of addition of Fe-Ti alloy on the microstructure of the sintered 13CrMoTi steels is shown in Photo. 4.

Fig. 4. Effect of addition of Fe-Ti alloy on tensile properties of sintered 13CrMoTi steels

Table 5. Effect of $\gamma\text{-Al}_2\text{O}_3$ addition on mechanical properties of sintered 13CrMoTi steel

$\gamma\text{-Al}_2\text{O}_3$ addition (%)	Sintered density (gr/cm ³)	0.2% proof stress (kg/mm ²)	Ultimate tensile strength (kg/mm ²)	Elongation (%)	Vickers hardness (10 kg load)
0	7.65	30.0	45.2	23.2	181
0.5	7.52	34.5	50.7	14.0	184
1.0	7.41	34.2	48.6	11.4	178

Photo. 4.

Change of microstructure of sintered 13CrMoTi steels by addition of Fe-Ti alloy ($\times 100$) (5/6)

- (a) Ti 0%
- (b) Ti 0.5%
- (c) Ti 2%

* The amount of Fe-Ti alloy added is referred to as Ti%.

Densification of the sintered compact was promoted and the porosity decreased considerably by addition of only 0.5% Ti. A further improvement in the sintering characteristics of these alloys was attained and the existence of pore in the product was scarcely observed with addition of 2% Ti.

Furthermore, the relationships between the tensile properties of the sintered AISI 406 steel with 0.5% of dispersed γ -Al₂O₃ and the amount of Fe-Ti alloy added are shown in Fig. 5. It is observed, in this case too, that addition of Fe-Ti alloy not exceeding 2% Ti is effective for improvement of the strength of the sintered compact, while it brings about only slight decrease in elongation. When the quantity of titanium reached 3%, the strength of the sintered compact slightly decreased. This is presumably because of the solid constituent of Fe-Ti alloy remained in grain boundary. Addition of Fe-Ti alloy showed the same effect for the improvement of the sintering characteristics of 13CrMoTi steel with dispersed γ -Al₂O₃.

Improvement of Strength of Sintered Compact by Hot Working

As characteristics of the dispersion strengthened alloys it has been said that the effect of the hot working is maintained for considerably a long period at elevated temperature as compared with conventional wrought materials.¹⁾ One reason for this fact is considered that a release of strain energy accumulated as a result of the hot working is very slow.^{1),3)} That is to say, a release of energy due to recovery or recrystallization does hardly occur with the consequence that the material is kept in as-worked condition and thus maintains the high strength for a long time.^{2),3)}

While the tensile strength of the alumina-dispersion strengthened steels was improved by means of the liquid phase sintering, some pores were still remained in the sintered compacts. Therefore, further investigation was performed in order to improve the mechanical properties of sintered specimens by means of hot working.

The test results on the sintered AISI 406 steel with 0.5% of γ -Al₂O₃ dispersed are shown in Table 6. In this case, the sintered specimens were hot-forged at the temperatures of 1100° to 900°C with forging ratio of about 1.5. As is clearly seen from this table, the hot-forging has little effect on the improvement of the tensile strength of the sintered specimen with no addition of Fe-Ti alloy. On the other hand, the strength of the sintered compacts with Fe-Ti alloy added is considerably high even in as-sintered state. Moreover, the hot-forging is markedly effective for the improvement of the strength of such sintered compacts. From these results, it can be said that a remarkable improvement of the strength of the dispersion strengthened steels concerned is attained by the combination of liquid phase sintering and hot working.

Figure 6 shows the tensile properties of the sintered AISI 406 steels with various amount of γ -Al₂O₃ with and without hot-forging. In this case, the quantity of Fe-Ti alloy added was 2% Ti, which gave the best result in Table 5. As for the strength at room tem-

Fig. 5. Effect of addition of Fe-Ti alloy on the tensile properties of sintered AISI 406 steels with dispersed γ -Al₂O₃ particles

Table 6. Effect of addition of Fe-Ti alloy and forging on tensile properties of sintered AISI 406 steel with dispersed 0.5% of γ -Al₂O₃

Ti addition (%)	Treatment	0.2% proof stress (kg/mm ²)	Ultimate tensile strength (kg/mm ²)	Elongation (%)	Reduction of area (%)
0	As-sintered	33.3	45.2	7.2	8.7
	Forged	33.7	48.2	13.6	15.3
1	As-sintered	39.9	50.8	6.0	4.0
	Forged	49.7	63.3	8.0	9.2
2	As-sintered	43.5	53.7	5.2	3.3
	Forged	59.6	71.2	4.8	4.4
3	As-sintered	38.5	49.5	4.6	2.8
	Forged	53.8	62.8	4.0	5.7

Fig. 6. Effect of hot forging on tensile properties of sintered AISI 406 steels (2% Ti addition)

perature, the increased addition of γ -Al₂O₃ from 0.5 to 1% resulted in its decrease, even though the hot forging was applied.

Table 7 shows the tensile properties at elevated temperature of various kinds of the sintered AISI 406 steels forged after sintering. When only γ -Al₂O₃ was added, the strengthening effect due to dispersed γ -Al₂O₃ was scarcely found. In contrast, when Fe-Ti alloy

was added together with $\gamma\text{-Al}_2\text{O}_3$, the elevated temperature strength of the sintered compact was greatly improved.

The reason for such improvement of the strength by addition of Fe-Ti alloy can be clearly understood from the electron microstructures shown in Photo. 5; in the case of $\gamma\text{-Al}_2\text{O}_3$ addition only, a remarkable coagulation of $\gamma\text{-Al}_2\text{O}_3$ occurred, while in the case of combined addition of Fe-Ti alloy, $\gamma\text{-Al}_2\text{O}_3$ particles were distributed nearly uniformly in grain boundary, although a considerable grain growth of them occurred, considering the initial particle size of $\gamma\text{-Al}_2\text{O}_3$ added.

That is, the high elevated temperature strength in the latter was due to the most favourable distribution of dispersed phase, while, in the former, the coagulated $\gamma\text{-Al}_2\text{O}_3$ would have a negative effect on high tem-

perature strength as impurities rather than dispersed phase.

From the above results, it is understood that Fe-Ti alloy not only improves the sintering characteristics but also improves the distribution of dispersed phase, thereby contributing to the increase of strengthening effect. So, in order to clarify the role of Fe-Ti alloy added during sintering, the analyses of aluminium and titanium which came from $\gamma\text{-Al}_2\text{O}_3$ and Fe-Ti alloy, respectively were performed, by using X-ray probe microanalyzer. The analytical results are shown in Fig. 7. In this case, pure iron powder was employed as the matrix substance in order to facilitate the analytical performance. The fact that titanium coexists with aluminium suggests that Fe-Ti alloy which turns to liquid state at the sintering temperature wets the surfaces of $\gamma\text{-Al}_2\text{O}_3$ particles at the first stage of sintering to separate the powders each other and redispense the powders coagulated during mechanical mixing of it with the matrix substance. Then some portion of Fe-Ti alloy will remain at grain boundary together with $\gamma\text{-Al}_2\text{O}_3$ after sintering.

The improvement of hot workability of the sintered compact is another effect of addition of Fe-Ti alloy. This could be certified by the fact that the sintered specimens with 0.5% of $\gamma\text{-Al}_2\text{O}_3$ were sometimes failed to be forged because of their poor hot workability, but when Fe-Ti alloy was added, the sintered compacts containing more than 1% of $\gamma\text{-Al}_2\text{O}_3$ could be satisfactorily forged.

The effect of a high forging ratio of about 6.5 on the mechanical properties of the sintered compact was examined. For this purpose, the sintered specimens, about 45 mm in diameter and 35 mm in height, sheathed in mild steel case of which thickness was 15 mm were forged.

The test results on the sintered AISI 406 steel are shown in Table 8. The results show that the tensile properties of the sintered compact can be further improved with increased forging ratio. With respect to the tensile properties of the sintered 13CrMoTi steel, the same tendency was also observed, except that the strength was slightly better than that of the sintered AISI 406 steel.

Table 7. Tensile properties of sintered AISI 406 steel at elevated temperature (650°C)
(Forged after sintering)

$\gamma\text{-Al}_2\text{O}_3$ addition (%)	Ti addition (%)	0.2% proof stress (kg/mm ²)	Ultimate tensile strength (kg/mm ²)	Elongation (%)	Reduction of area (%)
0	0	10.5	14.7	27.2	20.6
0.5	0	10.2	13.4	6.0	6.0
0.5	1.0	14.2	21.4	46.0	42.5
0.5	2.0	19.4	28.3	35.8	30.4

Photo. 5. Change of electron microstructure by addition of Fe-Ti alloy ($\gamma\text{-Al}_2\text{O}_3$: 0.5%, $\times 5\,000$) (5/6)
(a) Ti 0%
(b) Ti 2%

Fig. 7. Analytical results of Al and Ti by X-ray probe microanalyzer (Matrix: pure iron, $\gamma\text{-Al}_2\text{O}_3$: 1%, addition of Fe-Ti alloy: 2% as Ti)

Table 8. Effect of forging ratio on tensile properties of sintered AISI 406 steels
(γ -Al₂O₃ : 1%, Ti addition : 2%)

0.2% proof stress (kg/mm ²)	Ultimate tensile strength (kg/mm ²)	Elongation (%)	Reduction of area (%)	Forging ratio
48.0	59.8	4.2	4.1	1 : 1.5 (Hot forging)
53.2	67.1	7.2	7.8	1 : 6.5 (Sheath forging)
39.7	45.5	2.2	1.4	As-sintered

Photo. 6. Change of microstructure of sintered steels by difference of forging methods ($\times 300$) (5/6)
(a) Hot forging
(b) Sheath forging

Table 9. Example of tensile properties at elevated temperature (650°C) of dispersion strengthened steels
(γ -Al₂O₃ : 1%, Ti addition : 2%, sheath-forged)

Matrix	0.2% proof stress (kg/mm ²)	Ultimate tensile strength (kg/mm ²)	Elongation (%)	Reduction of area (%)
AISI 406 steel	22.5	32.2	14.4	18.4
13CrMoTi steel	24.2	34.2	33.8	57.9

Such an improvement of the tensile properties of the sintered compact by sheath forging is partly due to the work hardening by higher degree of hot working, but is considered to be largely attributable to the better densification of the compact. Photograph 6 shows the microstructures of longitudinal section of the sintered compact after forging. It is clear from these photographs that the sheath forged specimen has distinct fibre structure and the extent of densification in this specimen is in more favourable condition compared with that of the hot forged specimen.

Table 9 shows the elevated temperature tensile properties of the sintered specimens with 1% γ -Al₂O₃ and 2%Ti, sheath-forged after sintering. Compared with the results shown in Table 7, it is understood that the increased addition of γ -Al₂O₃ from 0.5 to 1.0% is more effective for improvement of the elevated temperature properties. Regarding the tensile properties at elevated temperature, the sintered 13CrMoTi steel is better in ductility as well as in strength than the 406 steel.

Fig. 8. Dimensions of test piece used for creep rupture test

Table 10. Comparison of creep rupture time
(Test temperature : 650°C, stress applied : 10 kg/mm²)

Matrix	γ -Al ₂ O ₃ (%)	Ti addition (%)	Creep rupture time (hr)	Remarks
AISI 406 steel	—	—	0.9	AISI 406 wrought steel
	0.5	—	1.98	Hot forging
	1.0	—	4.4	Hot forging
	1.5	—	2.8	Hot forging
	—	1.0	1.3	Hot forging
	—	2.0	1.5	Hot forging
	0.5	1.0	77.8	Hot forging
	0.5	2.0	181.4	Hot forging
	0.5	3.0	203.7	Hot forging
	1.0	2.0	284.6	Hot forging
	1.0	3.0	244.0	Hot forging
	1.0	2.0	1 177.1	Sheath forging
	2.0	2.0	148.5	Hot forging
	2.0	2.0	423.4	Sheath forging
	2.0	2.5	184.5	Hot forging
	2.0	3.0	377.2	Hot forging
	2.0	3.0	606.9	Sheath forging
13CrMoTi steel	—	—	3.5	Sheath forging
	0.5	—	8.8	Sheath forging
	1.0	—	19.9	Sheath forging
	0.5	1.0	141.9	Sheath forging
	1.0	2.0	3 973.2	Sheath forging
	1.5	3.0	342.5	Sheath forging

4. Creep Rupture Test Results

The methods of improving the tensile properties at room and elevated temperatures of 13Cr steels strengthened with dispersed γ -Al₂O₃ particles were established, as mentioned above. In order to evaluate the value for practical usage of this type of alloys as the heat resisting material, the creep rupture tests were carried out using smooth bar test specimens as illustrated in Fig. 8, using a multiple type creep rupture tester. Stress applied was adjusted by changing the diameter of the parallel part of the test specimen.

Table 10 shows the creep rupture strength of various sintered specimens. The test temperature and stress applied were 650°C and 10 kg/mm², respectively. The table also shows, for the sake of comparison, the test results on AISI 406 wrought steel. As can be seen in the table, the simple addition of γ -Al₂O₃ brings about only a slight improvement for the creep rupture property. In this case the creep rupture time can be

prolonged as the amount of $\gamma\text{-Al}_2\text{O}_3$ added is increased up to 1%. However, the extent of improvement is not so large. On the other hand, when Fe-Ti alloy is added together with $\gamma\text{-Al}_2\text{O}_3$, the creep rupture time is greatly prolonged. For example, the creep rupture properties of the hot forged sintered compact can be improved with the amount of $\gamma\text{-Al}_2\text{O}_3$ up to 2%, but the increase of the amount of Fe-Ti alloy added at the same time was further effective. On the other hand, the creep rupture strength could not be improved by the addition of Fe-Ti alloy only. The test results further reveal that the sheath forging of the sintered compacts improves their creep rupture strength to a large extent. The sintered 13CrMoTi steels having superior tensile properties at room and elevated temperature to the sintered AISI 406 steels are found to be better also in creep rupture properties.

Attempt was also made to improve the creep rupture properties of the sintered 13CrMoTi steel by increasing the amount of $\gamma\text{-Al}_2\text{O}_3$ added. The re-

sults, however, showed the fact that the addition of $\gamma\text{-Al}_2\text{O}_3$ over 1% decreased the creep rupture strength, even if the amount of Fe-Ti alloy was increased and

Fig. 9. Results of creep rupture test

AISI 406 steel after rupture			
13 CrMoTi steel before test			
13 CrMoTi steel after rupture			
Magnifi- cation	× 300 (5/6)	× 2000 (5/6)	× 5000 (5/6)

Photo. 7. Comparison of microstructure of sintered AISI 406 steel and sintered 13CrMoTi steels

sheath forging was performed. The sintering behaviour of 13CrMoTi steel is a little different from that of AISI 406 steel in this respect. This is probably because the liquid phase sintering by addition of Fe-Ti alloy failed to bring complete effect due to lower sintering temperature, 1 350°C.

Figure 9 compares the creep rupture properties at 650°C of the present two sintered steels with those of wrought steel having nearly the same chemical composition as AISI 406 steel powder used in this experiment and of Types 422 and 321 wrought steels as typical examples of martensitic and austenitic heat resisting steels, respectively. The creep rupture strength of AISI 406 wrought steel is very low and it decreases rapidly with the lapse of time. The creep rupture properties of the sintered AISI 406 steel with 1% of γ -Al₂O₃ dispersed was found to be slightly better than those of AISI 406 wrought steel, while the rupture strength of the sintered AISI 406 steels with dispersed γ -Al₂O₃ and Fe-Ti alloy, further processed by sheath forging, was very high and their rupture times at the same stress levels were much longer than those of the sintered specimens with addition of γ -Al₂O₃ only.

The rupture strength of the sintered and forged 13CrMoTi steels is found to be still higher than that of the sintered and forged AISI 406 steels, all the other conditions being the same. This superiority of the sintered 13CrMoTi steel may be partially attributed to the precipitation hardening of this steel.

Photograph 7 shows a comparison of microstructures of the sintered AISI 406 steel with that of the sintered 13CrMoTi steel, each of which contains 1% γ -Al₂O₃ and 2%Ti. A great number of fine precipitates, confirmed to be the intermetallic compound which has roughly the structure of Fe₂Ti as a result of the electron diffraction analysis, was found in the matrix of the sintered 13CrMoTi steels after creep rupture test, but such a microstructural change during creep rupture test was not seen in the sintered AISI 406 steel.

The short-time creep rupture strength of the dispersion strengthened 13CrMoTi steel is lower than that of Types 422 and 321 wrought heat resisting steel. However, the long-time creep rupture strength is superior to that of Type 422 steel, and it becomes higher than that of Type 321 steel after 5 000 hr, because of the small rate of lowering of the rupture strength as shown in Fig. 8. The sintered AISI 406 steel also shows comparable creep rupture strength to that of Type 321 austenitic steel after a certain long time.

As a result, these dispersion strengthened type of steels were found to be very promising as a new type of heat resisting material.

IV. Conclusion

In order to improve the elevated temperature properties, in particular, creep rupture strength of the ferritic steel, the fundamental studies on 13Cr steels were carried out. As a result, the dispersion strengthened type of alloys having superior elevated temperature properties could be obtained by means of the combination of liquid phase sintering and hot working. As an example, the long-time creep rupture strength of the sintered 13CrMoTi steel with dispersed γ -Al₂O₃ at 650°C was higher than that of Type 321 wrought steel. Consequently, this type of dispersion strengthened alloys are considered to be very promising as the new heat resisting materials.

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