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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HIP-CONSOLIDATED RENE 95 POWDERS

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Translation of "HIP ni yotte seikei shita Rene 95 no soshiki to kikaiteki tokusei," Journal of the Japanese Society of Powder and Powder Metallurgy, Vol. 25, No. 8, December 1978, pp 262-267

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16. Abstract

Effects of heat-treatments on the microstructure of P/M Rene 95 (a nickel-based powder metal), consolidated by the hot-isostatic pressing (HIP), were examined in some detail. The microstructure of as-HIP'd specimen was characterized by highly serrated grain boundaries. Mechanical tests and microstructural observations revealed that the serrated grain boundaries improved ductility at booth room and elevated temperatures by retarding crack propagation along grain boundaries.

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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HIP-CONSOLIDATED RENE 95 POWDERS

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I. Introduction /14*

Ni heat resisting alloys are used extensively in jet engines etc., and the heat resistant temperature has increased by more than 10°C annually for the past 30 years. Improvement ir such a characteristic has come about primarily because of increase in the type and amount of alloy elements used. Specifically, increaes in Al and Ti, which constitute the γ phase which is the principal strength element, and addition of large quantities of metals with high melting points such as Mo, W and Ta, contribute effectively to improvement in the mechanical properties of the alloy. However, increases in type and quantity of alloy elements invite deterioration in ductility and workability of the alloy, as well as unavoidable segregation in the casting state.

In order to eliminate such problems, the use of powder metals for production of heat resisting alloys has been considered. If alloy powders were used as the raw material, segregation of the alloy elements and non-uniformity of the microstructure would be restricted within the range of size of the fine powder. Ductility superior to that of cast materials would be anticipated. Consequently, the production of engine parts by powder metallurgy has flourished, especially in the United States.

* Numbers in the margin indicate pagination in the foreign text

However, there are many cases in the use of alloy powders as raw material in which the powder surface remains unchanged in terms of the grain boundary, even in the event of formation by forced working [1,2]. There are many cases in which oxide films of the powder surface remain in such grain boundaries [1], or in which dense arrangement of carbides occurs [1,2]. Consequently, the ductility of the material may not be as great as anticipated.

Various measures have been adopted to solve the problems originating in the properties of grain boundaries. An example would be the formation process in which the raw powder material does not contact air at all [3,4]. Here, removal of adsorbed gas or of inclusions in the powder has been attempted [4]. Moreover, various measures such as stabilization [5] of MC carbides have been adopted to reduce the amount of carbides on the grain boundaries.

The following measures were carried out as another attempt to improve the properties of the grain boundaries [5]. IN-792 alloy powder was extruded and air cooled after retention at the γ' solvus temperature. The γ' phase underwent nucleation on the grain boundaries during the course of cooling, and a shift in the grain boundaries occurred in the γ' phase. Ultimately, the grain boundaries assumed a wavy shape, resulting in improved ductility of the material. Analogizing from this, formation of wavy grain boundaries could be expected in the formation by hot-isostatic pressing (HIP) even if additional heat treatment were carried out. This is because the material would be gradually cooled within the equipment after retention at the HIP temperature.

In this research, the microstructure of Ni-based heat resisting alloy Rene 95 was studied in detail after it had gone through the standard forming process by HIP. Whether or not the grain boundaries assumed the desired wavy shape was confirmed. Changes in the shape of the grain boundaries in the case of /15 varied heat treatments, as well as changes in the resulting mechanical properties were examined.

Table 1 Chemical composition of René 95 powders (wt %).

Cr	Co	Mo	W	Ti	Al	Nb	Zr	B	C	Ni
12.9	8.2	3.3	3.4	2.5	3.3	3.4	0.06	0.01	0.05	Bal.

II. Experimental Method

Ar gas • atomized powder made by Kelsey Hayes Corporation was used as the raw powder. The chemical composition is shown in Table 1. The powder was spherical powder of 100 mesh granularity. The oxygen content was 80 ppm.

The powder was subjected to vibration compacting to a packing density of about 65% in a pyrex glass container for presintering. After heating in a vacuum to 540°C to remove the adsorbed gas on the powder surface, it was sealed in a vacuum of 3×10^{-5} Torr. The resulting powder which had been vacuum sealed was presintered for 10 hours at 1250°C. During cooling after sintering, the glass container broke due to the difference from the sintered material in the thermal expansion rate. This necessitated decanning.

After the aforementioned presintering, HIP treatment was carried out for three hours under conditions of 1180°C, 1000 atm pressure by hot isotactic press (peak temperature 1450°C, usual pressure 1300 atm, capacity 230 mmø x 1400 mm) model QIH 32, made by ASEA Corporation. After HIP treatment at this temperature, the sample was gradually cooled in the device, and the cooling rate at this time at about 1150°C was 3 to 5°C/min. This produced a

uniform sample 155 mm in diameter and 65 mm high with a theoretical density of virtually 100%.

Sections with a total length of 50 mm, length of 20 mm in the parallel sections and diameter of 2.5 mm were cut from the sample with the aforementioned configuration. After various heat treatments, including Rene 95 standard heat treatment[1], tensile tests and creep rupture tests were conducted. Tensile tests were carried out using Instron testers at room temperature and at 760°C. The tensile tests at 760°C were carried out in a vacuum. Conversely, the creep rupture tests were usually carried out in the air under stress of 55 kg/mm^2 at 760°C. Supplementary tests under the conditions discussed below were carried out on samples subjected to standard heat treatment.

Optical microscopic examination, replica observation and transmission electron microscopic examination were carried out to study the microstructural changes due to heat treatment. The thin film for transmission electron microscopic examination was prepared by electrolytic polishing using a solution of 10% perchloric acid and ethanol. Elution of the γ' phase under the conditions discussed below was carried out [6] to determine the volume fraction of the γ' phase in the sample. Electrolytic elution was carried out for four hours at a current density of 7 mA/cm^2 using an electrolyte of 1% ammonia sulfate + 1% citric acid. The elution residue was separated from the electrolyte by centrifugation, washed with water, dried and weighed.

III. Experimental Results and Considerations

[1] Rene 95 standard heat treatment conditions are 1093°C x 1 hr + QQ + 760°C x 16 hr. Heat treatment at 1093°C is the partial solution treatment. Here, the γ' phase does not completely become a solid solution.

Photo 1 illustrates the microstructure of the samples subjected to HIP treatment. The HIP temperature of 1180°C was higher than the γ' phase solvus temperature (about 1150°C [7]), but the γ' phase precipitated in the particles and on the grain boundaries during the course of cooling after HIP. The γ' grains in the particles were cubes 0.3~0.4 μ on a side. γ' phase larger than that formed on the grain boundaries. The volume fraction of the γ' phase in this state was about 40%.

The characteristic of the microstructure shown in Photo 1. is that the grain boundaries are not smooth, but that they have waviness of several μ. The characteristic shape of such grain boundaries is retained even during partial solution treatment carried out for 1 hour at 1093°C after HIP. Photo 2 illustrates the microstructure in this state. A comparison of this with the HIP microstructure of photo 1 reveals that the γ' phase in the particles underwent partial solution treatment due to heat treatment at 1093°C, resulting in scattering, but the mean particle diameter remained virtually unchanged. The volume fraction of the γ' phase in this state was about 28%. The microstructure of samples subjected to such heat treatment will henceforth be termed "standard".

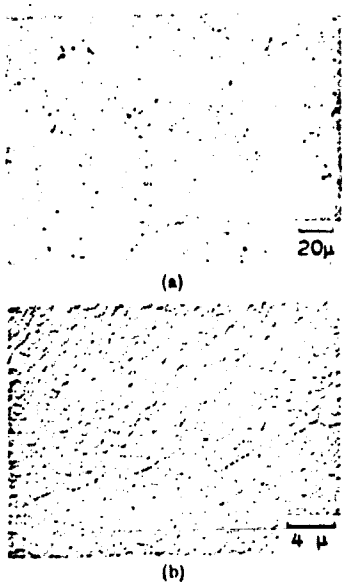


Photo. 1 Microstructures of as-HIP'd René 95.

The wavy grain boundaries formed by HIP treatment is smooth, due to complete solution treatment of the γ' phase. Specifically, as illustrated in Photo 3, the γ' phase undergoes solution treatment, and the grain boundaries /16

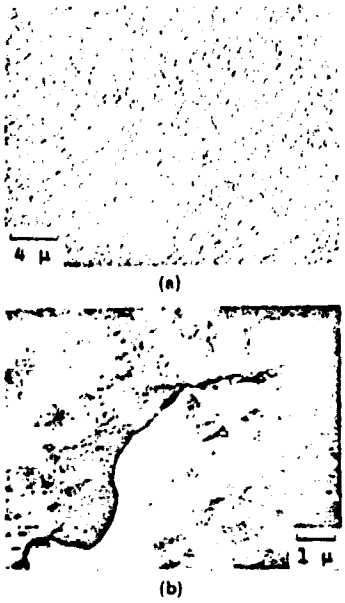
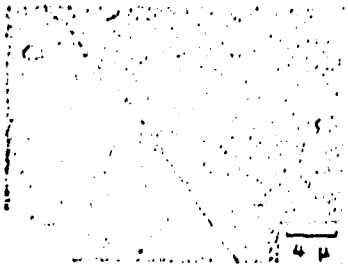


Photo. 2 "Standard" structures of René 95.

become smooth when heat treatment is carried out for 1 hour at 1170°C after HIP followed by quenching. The γ' phase in the particles also should undergo complete solution treatment at 1170°C. The fine γ' particles in the granules seen in the photograph have precipitated during the course of cooling because the quenching operation after heat treatment was incomplete.

The grain boundaries which became smooth due to heat treatment at 1170°C were gradually cooled from this temperature across the temperature at which precipitation of the γ' phase begins, resulting in a curved form again. Photo 4 illustrates the microstructure of the sample gradually cooled at the rate of 7~8°C/min to 1120°C after retention for one hour at 1170°C. According to this figure, the grain boundaries serrated by the γ' particles which precipitated during gradual cooling were curved between the granules. Samples with the essentially identical microstructure were observed following gradual cooling from 1170°C to 1150°C. This indicates that precipitation of the γ' phase begins from temperatures somewhat higher than 1150°C.



structure of René 95 heat-treated at
after HIP.

The above observation results clarify that the curved grain boundaries in the sample subjected to HIP treatment are formed during gradual cooling after HIP.

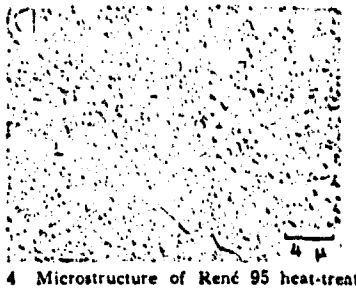


Photo. 4 Microstructure of René 95 heat-treated at 1170°C and slowly cooled down to 1120°C.

When samples are subjected to heat treatment at 1170°C after HIP treatment, and then gradually cooled to 1093°C at the same cooling rate as that employed before, oil quench hardening results in a precipitated state of the γ' phase in the particles which is virtually identical to the "standard" microstructure shown in Photo 2. Specifically, the length of a side of the γ' granules in the particles is 0.3~0.4 μ, as shown in Photo 5. Moreover, the total volume fraction of the γ' phase is about 30%. The grain boundaries are curved identically to the "standard" case, but the γ' phase precipitated in the grain boundaries exhibits longer fine elongation than in the "standard" case. The microstructure shown in Photo 5. is termed "wavy" below.

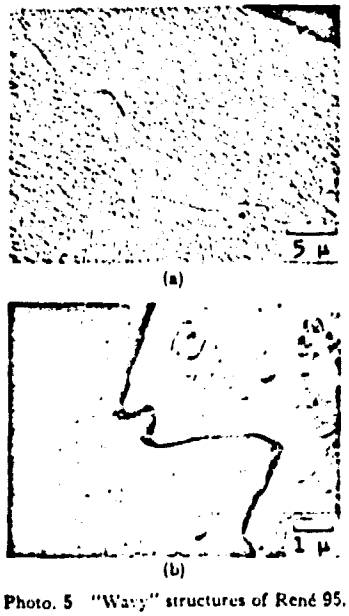


Photo. 5 "Wavy" structures of René 95.

The cooling rate traversing the temperature at which precipitation of the γ' phase begins determines the extent of waviness of the grain boundaries. The reason is that when the cooling rate exceeds a given level, dense nucleation of the γ' phase occurs before shift of the grain boundaries induces waviness, resulting in fixation of the grain boundaries. Actually, when the cooling rate from 1170°C is 25°C/min, there is only slight waviness of the grain boundaries.

/17

When a sample is air cooled after heat treatment for one hour at 1170°C, and then reheated to 1150°C in a furnace, nucleation of the γ' granules densely occurs along the grain boundaries during the course of temperature rise to 1150°C. Consequently, the grain boundaries are smooth even if gradually cooled following retention at 1150°C. Photo 6 illustrates that microstructure. In this case, the sample was held at 1150°C for 15 minutes, then gradually cooled at the slow rate of 2~3°C/min to 1093°C, and then subjected to oil quench hardening. When such heat treatment is carried out, equiaxial γ' granules densely form on the smooth grain boundaries. Photo 6(b) indicates that the grain boundaries are as wavy as the size of the γ' particles, but this structure is macroscopically smooth. In this case, the volume fraction of the γ' phase is approximately 28%. The size of the γ' granules in the particles is about 0.3~0.4 μ. Specifically, the precipitation state of the γ' phase in the particles is virtually identical to "standard" and "wavy". Such a microstructure is henceforth termed "smooth".

Table 2 illustrates the relation between microstructure and the three types of heat treatment conditions discussed above. In these microstructures, the amount of γ' phase and the size of the γ' granules in the particles are virtually identical, but the shape of the γ' granules precipitated on the grain boundaries and the shape of the grain boundaries differ.

Next, the mechanical properties were studied on samples subjected to standard aging treatment for 16 hours at 760°C following the aforementioned three types of heat treatment. Fine γ' granules precipitated in the particles due to this aging treatment. For that reason, the volume fraction of the γ' phase in "standard" samples, for instance, is about 40%. The mean particle diameter of such fine γ' granules was about 500 Å even after 200 hours of creep deformation at 760°C.

Table 2 Characteristics of microstructures after var[illegible]t-treatments.

	Heat treatment after HIP	Size of γ' [illegible]	Volume fraction of γ'	Morphology of γ' on G.B.
Standard	1093°C × 1 hr/OQ	0.3~0.4	28%	blocky
Wavy	1170°C × 1 hr $\xrightarrow{SC}$ 1093°C/OQ	0.3~0.4 μ	30%	elongated
Smooth	1170°C × 1 hr/AC ⟶ 1150°C × 15 min $\xrightarrow{SC}$ 1093°C/OQ	0.3~0.4 μ	28%	equiaxial

OQ: oil quenched SC: slowly cooled AC: air cooled

Table 3 Mechanical properties of HIP'd and heat-treated René 95.

	Tensile properties at 760°C			Creep-rupture properties at 760°C, 55 kg/mm²	
	Proof stress (kg/mm²)	UTS (kg/mm²)	Elongation (%)	Rupture life (hr)	Elongation (%)
Standard	104	108	9.1	219	7.4
Wavy	102	109	8.0	148	5.5
Smooth	100	108	6.4	127	4.9

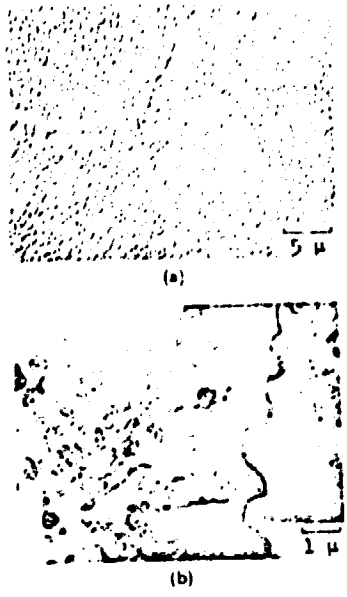


Photo. 6 "Smooth" structures of René 95

Table 3 illustrates the results of tensile tests at 760°C and of creep rupture tests conducted under stress of 55kg/mm^2 at 760°C.

The tensile strength at 760°C exhibited virtually no change based on the heat treatment conditions. This is a natural result of the absence of differences in precipitation states of the γ' phase in the particles. The elongation values were best for the "standard" samples, and worst for the "smooth" samples. The tensile characteristics at room temperature were virtually constant, regardless of the heat treatment conditions. The proof stress was about 116 kg/mm^2; the upper tensile strength was about 146 kg/mm^2 while the elongation rate was about 12%.

The differences between the three types of heat treatment are distinct with regard to the creep rupture characteristics. Specifically, both the life and elongation are best in "standard" samples and are worst in "smooth" samples. /18

Photo 7 illustrates the cracks after creep rupture at 760°C. This photograph clearly indicates that cracks are easily propagated along the grain boundaries in "smooth" samples, while the direction of crack propagation in "standard" samples is curved due to waviness of the grain boundaries. This indicates that the propagation of cracks is blocked in "standard" samples. Consequently, their creep rupture characteristics are good. The inferiority of the creep rupture characteristics of "wavy" samples to those of "standard" samples is due to the ease of propagation of cracks along the elongation of the γ' phase along the grain boundaries.

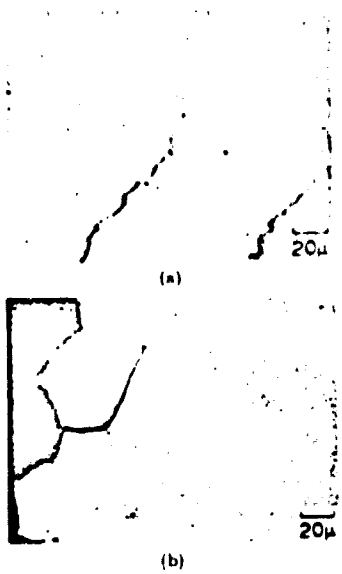


Photo. 7 Micrographs showing propagation of cracks in René 95 during creep deformation.
(a): "standard" (b). "smooth"

Figures 1 and 2 compare the mechanical properties of "standard" samples with the characteristics of sintered material (P/M material)[8] reported by other researchers as well as with those of cast + wrought materials [8,9]. The characteristics of the P/M materials shown here are for materials wrought at high temperature following HIP treatment. The result is a microstructure termed a necklace structure. In this case, extremely fine recrystallized particles form along the periphery of the grain boundaries in the stages of HIP. Consequently, the transposition movement due to deformation occurs uniformly, and the ductility improves [10].

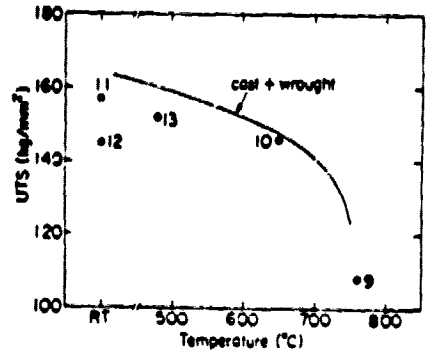


Fig. 1 Comparison of UTS values of "standard" specimens (○) with those of P/M (●)[9] and cast + wrought René 95[9]. Values of elongation are presented for each test.

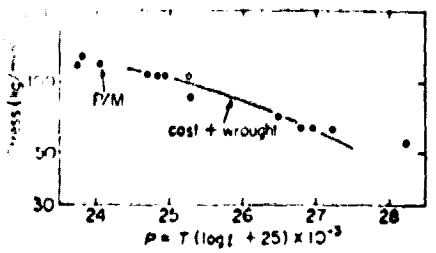


Fig. 2 Comparison of creep-rupture properties of "standard" specimens (○) with those of P/M (●)[9] and cast + wrought René 95[9]. The values of creep stress are plotted against the Larson-Miller parameter $P = T\ (\log t + 25)$ where T is the test temperature and t is the rupture life.

According to Fig.1, the upper tensile strength of "standard" samples determined in this research is somewhat inferior to the characteristics of P/M materials determined by other researchers. They do not reach the level of the characteristics of smelted materials. A detailed examination of the heat treatment conditions is necessary for improvement of the tensile characteristics of materials formed by HIP.

Fig.2 demonstrates that the creep rupture characteristics of "standard" samples are better than those of P/M samples according to other researchers as well as better than those of smelted materials. In this figure, in addition to the characteristics at 760°C discussed previously, the characteristics under conditions of 105 kg/mm^2 at 649°C and 63 kg/mm^2 at 704°C are illustrated as characteristics of "standard" samples. The elongation level at 649°C and 704°C was 6~7% in both cases. According to Barker et al. [8], the elongation of P/M material with a necklace structure was 5% lower under conditions of 105 kg/mm^2 at 649°C. This indicates that the characteristics of the "standard" material are superior.

IV. Conclusions

The effects of heat treatment on the microstructure and strength characteristics of Rene 95 formed by HIP have been studied, with the following conclusions.

1) A characteristic in the microstructure of samples subjected to HIP is a waviness of several μ in the grain boundaries. Serration of these grain boundaries occurred due to the γ' phase during gradual cooling of the samples in the equipment following HIP. These shapes formed due to shift of the grain boundaries during serration. /19

2) The grain boundaries become smooth due to solution treatment of the γ' phase of the grain boundaries when the samples subjected to HIP treatment were quenched at the γ' phase solvus temperature. However, when gradual cooling was carried out traversing the temperature at which precipitation of the γ' phase begins, the grain boundaries again become wavy.

3) Samples with smooth grain boundaries and samples with wavy grain boundaries can be formed by selection of various heat treatment conditions, but samples are produced in which the shape of the γ' phase precipitated on the grain boundaries becomes longer and finer than that of samples merely subjected to HIP. A comparison of the mechanical properties of samples with these varying microstructures reveals conspicuous differences in the tensile elongation at 760°C and in the creep rupture characteristics. The best characteristics were achieved by conducting standard heat treatment after HIP, while the characteristics of samples with smooth grain boundaries were worst. This difference is related to the ease of crack propagation along the grain boundaries.

4) The creep rupture characteristics of samples subjected to standard heat treatment after HIP are better than those of P/M (samples formed with a necklace structure due to high temperature forging) material reported by other researchers as well as better than those of smelted material.

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