PM HIP NNS Process for Valve Bodies Made of Grade 91 Material

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Abstract

Shimoda Iron works Co., Ltd. and Metal Technology Co. Ltd. are developing PM HIP NNS process for pipe fittings made of high grade materials. As a first trial for full scale manufacturing, valve bodies for thermal power plant made of 9Cr-1Mo material are being manufactured. After HIPing and heat treatment some metallurgical and mechanical investigations were made. The results show uniform characteristics and good strength. This paper introduces an outline of the PM HIP NNS procedure and the investigation results.

1. Introduction

The PM HIP process (Powder Metallurgy - Hot Isostatic Pressing process) is a state-of-the-art technology to manufacture metal products made of high grade materials, such as, stainless steels and super alloys[1)]. This process is not casting nor forging but a kind of powder sintering.
Shimoda Iron Works Co., Ltd. (herein after “Shimoda”) is fundamentally a producer of forged fittings for piping however Shimoda is now developing a new process to manufacture fittings using this PM HIP process in collaboration with Metal Technology Co. Ltd. (herein after “MTC”) who specializes in HIP treatment.
As an initial trial of this new process, a type of valve body for thermal power plants was selected to be manufactured. The material of this product is 9Cr-1Mo also known as “Grade 91” which has high strength at high temperature condition. In this paper, the trial production and some of the investigation results of the product will be reported.

2. Outline of HIP treatment

2.1 HIP unit

Figure 1 shows the basic construction of a HIP unit. There are electric heating coils in a pressure vessel and high pressure Argon gas is charged in it. The heating temperature is higher than 1,000℃ and the pressure is more than 100MPa. The gas pressure acts uniformly in all directions therefore the products can be pressed isostatically. Owing to this isostatic pressing, the product displays uniform characteristics throughout.

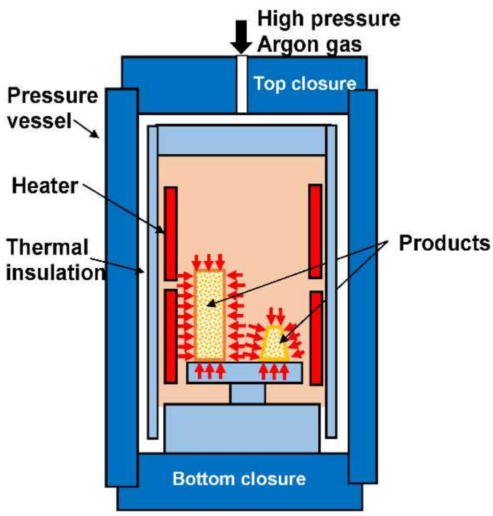

Figure 1 HIP unit

2.2 PM HIP NNS process

There are several applications for HIP units, such as, densification of cast parts or 3D printed parts, diffusion bonding between similar and dissimilar metals, sintering of metal powder to name a few. Among these applications, PM HIP NNS process will be the focus of this paper. This process is kind of a metal powder sintering process however the special feature of this process is that the metal powder fills the capsule before HIP treatment. The capsule is prepared depending on the dimensions of the final product and therefore enabling a Near Net Shape blank to be obtained after HIPing. NNS HIPing reduces both metal powder costs and finish machining costs.

When applying the PM HIP NNS process the design of the capsule and the dimensions are critical. Since the powder consists of spherical grains, there are some spaces between grains in the capsule before HIPing. The density before HIPing is around 65-70%. However HIP treatment increases the density up to 100%. This results in more than 30% shrinkage in volume taking place. As a consequence the capsule designer has to be able to predict the shrinkage cased by HIPing. This is one of the key points to successfully being able to carry out the PM HIP NNS process.

2.3 Benefits of PM HIP NNS process

The main benefits of the PM HIP NNS process are as follows:

1) Flexibility of chemical composition design, especially alloy contents.
2) Uniform characteristics in the whole section and in all directions.
3) Elimination of welding. It can not only reduce the production cost but also solve the problem of weak strength performance and corrosion resistance at the welded areas.
4) Reduction of material costs and finish machining costs.
5) Large and more complex shapes can be manufactured.

3. Valve body production

3.1 Product designation

A type of valve body which is designed for thermal power plants was selected for the trial production by PM HIP NNS process. Conventionally these valve bodies have been manufactured by a forging process.

3.2 Material

The material of the valve body is UNS K90901 (9Cr-1Mo-0.2V-Nb-N) which is specified in ASTM A989 for HIPed alloy steels[2)]. This material is one grade of so called 9Cr materials which has a high strength in high temperatures. Since this material is the equivalent to ASTM A182 F91 which is specified for forged products this is called “Grade 91” in this paper.

Table 1 shows the chemical composition of the powder material and **Figure 2** shows the SEM observation of the powder. The grain size is 2-90μm.

Table 1 Chemical composition (wt%)

		C	Mn	Si	Cr	Ni	Mo	V	N	Nb
A989 K90901	min.	0.08	0.30	0.20	8.0	-	0.85	0.18	0.03	0.06
	max.	0.12	0.60	0.50	9.5	0.40	1.05	0.25	0.07	0.10
Powder		0.10	0.39	0.40	8.77	0.17	0.90	0.25	0.05	0.08

3.3 Capsule preparation

Figure 3 shows the capsule configuration before and after HIPing. The powder density is predicted to be 67%. The black square area is a mild steel block which is necessary to fabricate the capsule by welding. This mild steel block is eliminated by vertical drilling at the post machining stage.

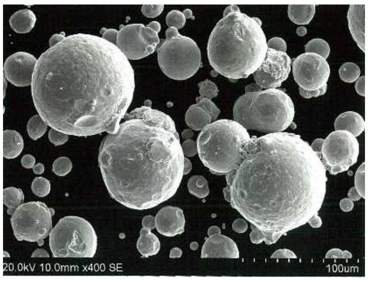

Figure 2 SEM photograph of the powder

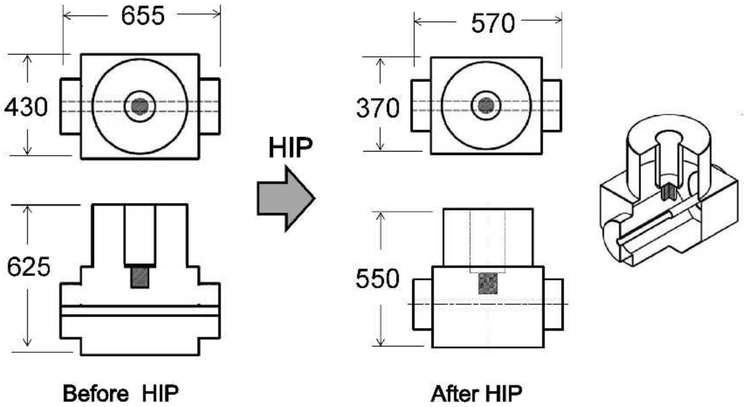

Figure 3 Capsule design before and after HIP

3.4 HIP treatment

After the capsule filling and de-gassing, the inlet nozzles are sealed. **Figure 4** shows the capsule ready for HIPing. HIP treatment is carried out in the Giga HIP unit at MTC. The Giga HIP is the world's largest working zone (2,050mm dia. x 4,200mm height)..

3.5 Removing capsule and heat treatment

After removing the capsule by machining the part then underwent the following heat treatment.

Quenching: 1,050℃ x 3hr → oil quenching
Tempering: 760℃ x 4.5hr → cooling in still air

Figure 5 shows the product after heat treatment.

Figure 4 Before HIP

Figure 5 After HIP and heat treatment

4. Investigations and evaluation

4.1 Investigation items

The following investigations were carried out:

1) Tensile tests at room temperature
2) Brinell hardness distribution
3) Tensile tests at high temperature up to 700℃
4) Impact tests (Charpy V notch at room temp.)
5) Macrographic observations of the section
6) Micro structures
7) Grain size measurements
8) Non-metallic inclusion
9) Creep tests

4.2 Tensile tests at room temperature

Tensile tests are made at various locations and in every directions. As shown in **Figure 6**, all the results show uniformity at all locations and directions.

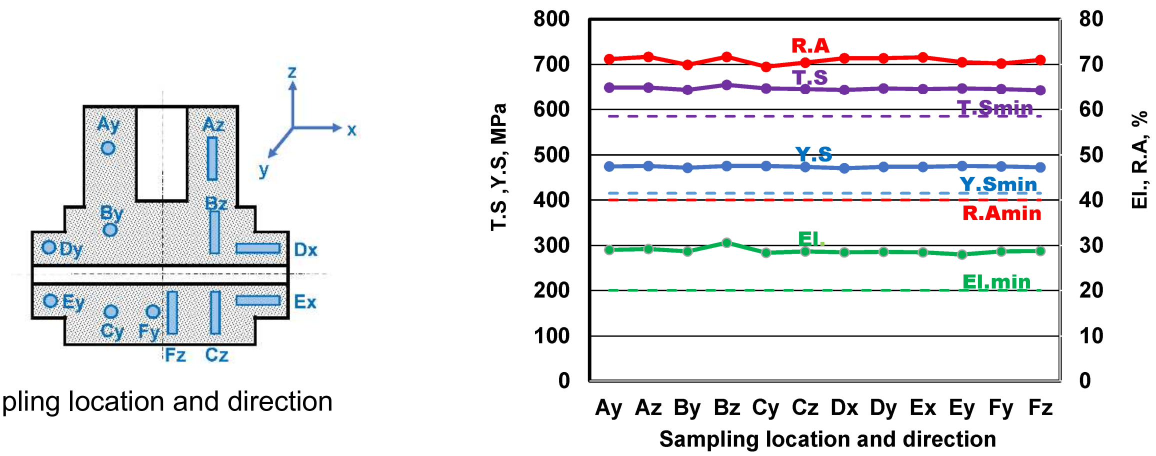

Figure 6 Tensile test results

4.3 Brinell hardness distribution

Figure 7 shows the Brinell hardness distribution of the whole section. Hardness values are uniform (HB200±5) with the exception of the mild steel area.

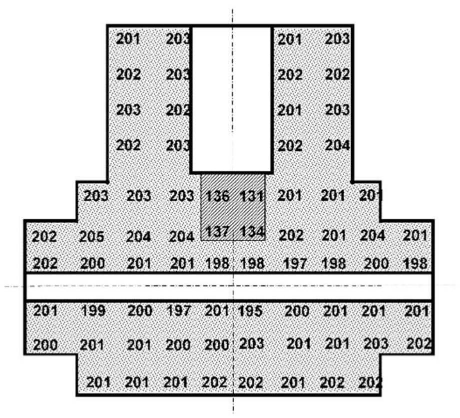

Figure 7 Brinell hardness distribution

4.4 Tensile tests at high temperature up to 700℃

The tensile strengths for the high temperature tensile tests from 100℃ to 700℃ are shown in **Figure 8** together with a results for the room temperature (20℃). Reference line in the figure shows the data of Grade 91 material reported by EPRI[3)] (Electric Power Research Institute). The results are greater than this line.

4.5 Impact tests (Charpy V notch at room temp.)

Figure 9 shows the results of the impact tests at room temperature. Firstly there is less variation between the three individual results at the same condition compared with our usual experiences on forged products. Moreover there is little variation among the average values at any of the locations and in any direction. This uniformity of impact characteristics is due to the uniformity and the fine grain size as reported later.

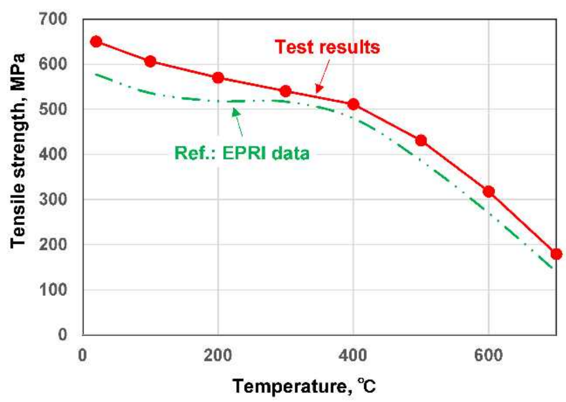

Figure 8 High temperature tensile test results

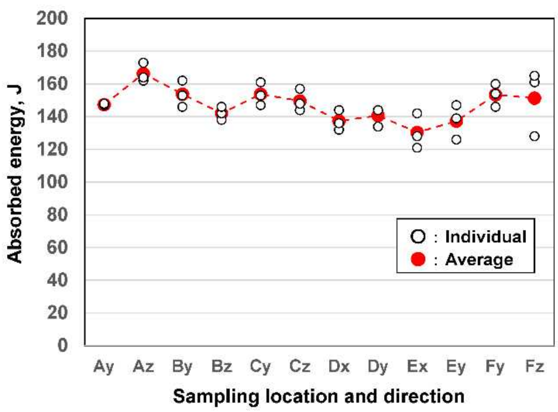

Figure 9 Impact test results (at room temperature)

4.6 Macro structure

Figure 10 shows the macro structure of the section. It is reasonably uniform, no segregation nor voids. Needless to say that there no metal flow is observed.

4.7 Micro structure

Every micro structure is almost the same at any location. **Figure 11** shows an example of micro structure at the middle of the section.

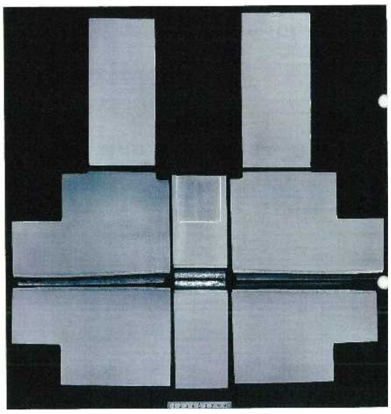

Figure 10 Macro structure

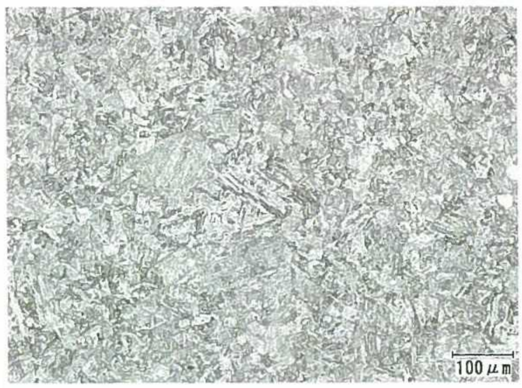

Figure 11 Micro structure

4.8 Grain size

Grain sizes are measured at 12 points of the section. As shown in **Table 2**, the grain sizes are remarkably fine at every location.

Generally speaking, the grain sizes of forged products are much coarser than these results and as a result the grain sizes of a forged part vary widely depending on the locations. The size in the middle area is usually coarser than the surface area. In stark contrast the PM HIP part's most noticeable characteristic is the uniformity and fine grain size.

4.8 Non-metallic inclusion

Non-metallic inclusions are investigated at several points of the section.　As shown in **Table 3,** only D series inclusions (globular oxide) are observed. These results means the material has good cleanliness.

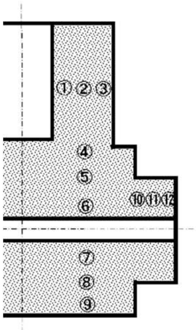

Table 2 Grain size

Location	①	②	③	④	⑤	⑥
Grain size	10	10	10	10	10	10
Location	⑦	⑧	⑨	⑩	⑪	⑫
Grain size	10	10	10	10	9.5	9.5

Table 3 Non-metallic inclusions

A		B		C		D	
Sulfid		Alumina		Silicate		Globular oxide	
Thin	Thick	Thin	Thick	Thin	Thick	Thin	Thick
0	0	0	0	0	0	0.5-1.0	0.5

4.9 Creep tests

Creep tests have been made on the test pieces sampled from the product. The results are shown in **Figure 12**. All specimens were ruptured. The longest test duration is approximately 22,000 hours (2 years and 6 months) at 600℃.

The lines in the figure are reference lines reported by NIMS (National Institute for Material Science) which are showing the conventional test results on existing Grade 91 materials. The solid line means the average of the conventional data and dotted line means 99% bottom limit respectively. According to the test results and data by NIMS, it can be concluded that the valve body has the equivalent creep strength to conventional Grade 91 materials.

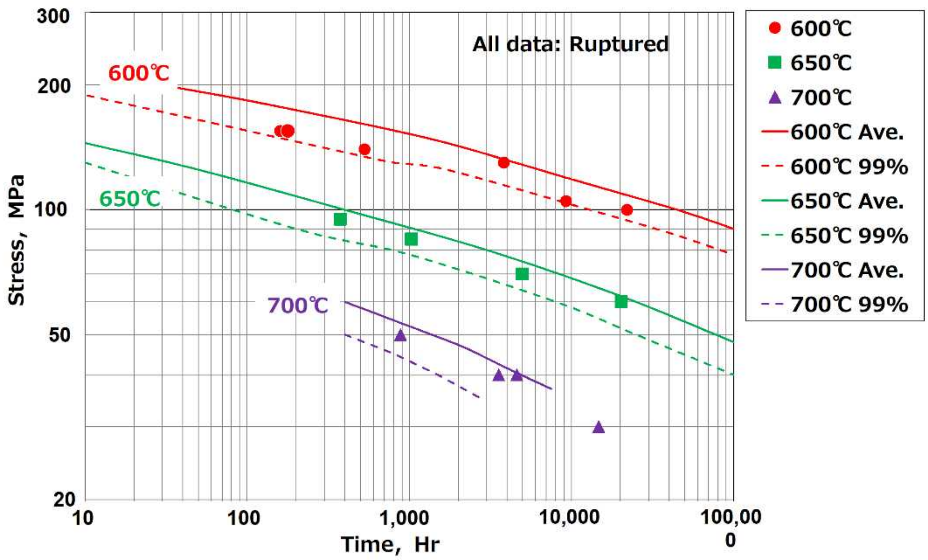

Figure 12 Creep test results

5. Finish machining

The blank shown in Figure 5 is finished by Shimoda's machining factory. The finished valve body is shown in **Figure 13**. If this valve body is manufactured through a forging process, it is necessary to forge a mono block blank of approximately 900kg. On contrast, the HIPed NNS blank is only 536kg which is around 40% less than the forged blank. Also, the finish machining time of the forged mono block blank is estimated to be around 100 hours. However the finishing time for the HIPed blank is only 70 hours which is 30% reduction compared to the forging process.

Figure 13 Finished valve body

6. Conclusion

1) Valve bodies for a thermal power plant made of Grade 91 material can be successfully manufactured by the PM HIP NNS process.
2) Due to experienced design of the capsule dimensions, the HIPed product has NNS configuration for finish machining.
3) Thanks to the NNS configuration, the weight of a blank and the finish machining times are reduced up to 40% and 30% respectively compared to the forging process.
4) The investigation results show satisfactory characteristics of the HIPed product. The most notable feature is the uniform characteristics at any location and in any direction.
5) Creep test results have proven that the product has the equivalent creep strength to conventional Grade 91 material.

References

1) Introduction to PM HIP Technology, EPMA (European Powder Metallurgy Association) 2011 2nd edition.
2) ASTM A989/989M Standard Specification for Hot Isostatic Pressed Alloy Steel Flanges, Fittings, Valves, and Parts for High Temperature Service.
3) Powder Metallurgy / Hot Isostatic Processing (PM/HIP) Data Package for Grade 91,EPRI