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Consolidation process study of 9Cr-ODS martensitic steels

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Abstract

As a practical method to produce a large-scale annular billet made of 9Cr-ODS martensitic steels, a hollow capsule with outer and inner diameter of 147 and 32 mm, respectively, was filled with mechanical milled (MM) powders and were extruded by a 2000 ton press at 1423 K. Hot isostatic pressing was applied as a consolidation process of MM powders at 1423 K and 190 MPa for 3.6×10^3 s. The microstructure and formability of the HIPed product were investigated. The hot-extrusion behavior of the HIP-ODS alloy was also tested using a 2000 ton press. The compaction by the cold isostatic pressing with subsequent sintering is insufficient as a starting material for hot-extrusion.

1. Introduction

The 9Cr-ODS (oxide dispersion strengthened) martensitic steels should be prospective materials not only for the long life fuel cladding of the liquid metal fast reactors [1,2] but also for the low activation fusion reactor [3], due to high temperature capability as high as 973 K. The typical 9Cr-ODS martensitic steels have been developed so far for fuel cladding application in the Japan Nuclear Cycle Development Institute [4]. The manufacturing process is, however, limited to small scale production. In order to apply 9Cr-ODS martensitic steels as claddings of the driver fuel pins in the Japanese prototype first breeder reactor MONJU, economical mass production will be required in both processes of consolidation of mechanical milled (MM) powders and cold-rolling of claddings. Regarding the latter process of cladding manufacturing, cold-rolling process using two rolls type of pilger mill was applied, and its technology has been almost accomplished as an applicable mass-production method to manufacture 2 m length of 9Cr-ODS martensitic steels claddings. On the other hand, consolidation method of annular billet is currently insufficient; annular billets are manufactured by mechanically drilling the extruded bars and by grinding the outer surface to the 18 mm in diameter and 3 mm in thickness. This method is disadvantageous for manufacturing the long length annular billets for mass production.

In this study, a practical process for consolidation of the MM powders is investigated with respect to the technology development of large scale production of the annular billet.

2. Hollow capsule extrusion

The annular billet can be directly manufactured without drilling the hole by hot-extrusion of hollow capsule in which MM powder is filled. The chemical composition of the MM powder used in this experiment is 9Cr–0.14C–2W–0.2Ti–0.35Y_2O_3, which is shown in Table 1. These powders were manufactured by means of an attrition type ball mill with milling period of 1.73×10^5 s. The hot-extrusion experiment was conducted by using 400 ton hydraulic press with container of

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Table 1
Chemical composition of MM powders (mass%)

C	Si	Mn	P	S	Ni	Cr	W	Ti	Y	Y_2O_3	O	Ex.O	N	Ar
0.14	<0.01	<0.01	0.002	0.003	<0.01	9.00	1.92	0.20	0.28	0.36	0.15	0.07	0. 0092	0.0028

67 mm diameter, and the large scale of 20,000 ton hydraulic press was also applied for hot-extrusion to evaluate production capability of the real size annular billet.

2.1. 400 ton press experiment

The dimension of the hollow capsule used is 67 and 20 mm in outer and inner diameter, respectively. The inner wall of the hollow capsule is made of 17Cr stainless steel, which corresponds to the liner of cladding after cold-rolling of the annular billet to maintain better compatibility with a fuel as a fuel pin cladding. The hollow capsule was filled with MM powders of 2 kg weight, then sealed by the tungsten inert gas welding. The hot-extrusion test was conducted at 1423 K using 400 ton press, which consists of a die of 30 mm diameter and mandrel of 18 mm diameter. These dimensions correspond to the outer and inner diameter of the produced annular billet with about 1 m in length. The test condition consists of extrusion speed of 30 mm/s and extrusion pressure of 616 MPa. The extrusion ratio, which is a ratio of cross-section before and after extrusion $(67^2 - 20^2)/(30^2 - 18^2)$, becomes a value of 7. By the direct extrusion of the hollow capsule, target dimension of the annular billet was attained, and uniformity in the thickness of the consolidated 9Cr-ODS ferritic steel parts was maintained along the axial direction of the annular billet.

2.2. 2000 ton press experiment

In order to study feasibility of economical mass production of large size of annular billet, hot-extrusion test was conducted using large scale of 2000 ton press. The dimension of the hollow capsule is 147 and 32 mm in outer and inner diameter, respectively. The total weight of MM powders reaches 30 kg. An appearance of large hollow capsule is shown in Fig. 1. The manufactured annular billet size is 48.9 mm in outer and 28 mm in inner diameter and 3 m in length. An extrusion ratio, $(147^2 - 32^2)/(48.9^2 - 28^2)$, corresponds to a value of 13. Fig. 2 shows the cross-section of the manufactured annular billet. The inner wall is made of 17Cr ferritic stainless steel, and it becomes liner of the inner surface of the cold-rolled claddings. The outer wall, however, should be mechanically removed from the consolidated ODS martensitic steel. This additional process of outer wall removal may raise cost. The annular billet having above dimension has a capability providing about 100 pieces of typically commercialized claddings with 8.5 mm in outer diameter, 0.5 mm in thickness and 2 m in length, which are generally produced by cold-rolling process.

Fig. 1. Photograph of a large-scale hollow capsule. Outer diameter: 147 mm and inner diameter: 32 mm.

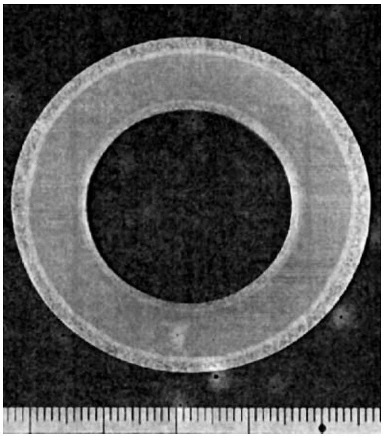


Fig. 2. Cross-section of manufactured annular billet with inner wall tube made of 17Cr stainless steel. Outer diameter: 48.9 mm and inner diameter: 28 mm.

3. HIP-extrusion process

The process of annular billet production from the hollow capsule by hot-extrusion as mentioned in the previous section requires a tightly welded capsule to maintain integrity of the filled MM powders during hot-extrusion at 1423 K. The manufacturing of adequate capsule is expensive. A consolidation process made by hot isostatic pressing (HIP) has advantage, since its hot-extrusion is easier and capsule needs less tight structure. For this purpose, feasibility of HIP-extrusion process was investigated using the 9Cr–0.14C–2W–0.2Ti–0.35Y_2O_3 powders (Table 1).

3.1. Properties of HIP-product

Billet in 160 mm diameter was produced by HIP processing at 1423 K with a pressure of 190 MPa for 3.6×10^3 s that is almost upper condition for the temperature of the 9Cr-ODS martensitic steels and for the pressure capability of the equipment. An optical micrograph is represented in Fig. 3. Many black dots like pores remain at the boundaries of the original powder. The high temperature and high speed tensile test was conducted for the HIPed product. Figs. 4 and 5 exhibit the tensile strength and reduction of area as a function of temperature, respectively. Tensile strength of HIPed product in 9Cr-ODS martensitic steel is approximately four times higher than that of a typical austenitic 304 stainless steel at 1423 K, and so the deformation resistance of HIPed product is expected to be significantly high during hot-extrusion process. The value of reduction of area in HIPed product decreases with increasing temperature: inverse temperature dependency to that of usual stainless steels. This reduction of area of HIPed product, which is about 1/3 times lower than 304 stainless steel at 1423 K, leads to less formability and hence insufficient to directly make claddings by cold-rolling from HIPed product. Based on these results, property improvement of HIPed product by hot-extrusion should be indispensable in the course of cladding manufacturing.

3.2. Hot-extrusion tests

Using a 400 ton press, HIPed products in 67 mm outer diameter were hot-extruded at the extrusion ratio of 3, 4 and 6 at 1473 K. The relationship between measured extrusion pressure vs. extrusion ratio is shown in Fig. 6. There is linear relationship between extrusion

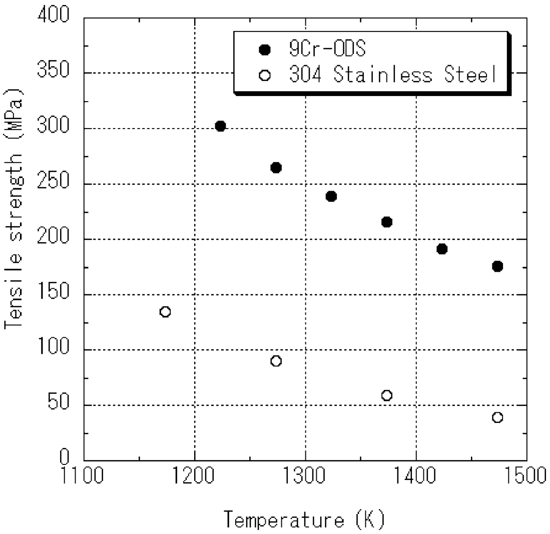


Fig. 4. Temperature dependency of tensile strength of HIPed product.

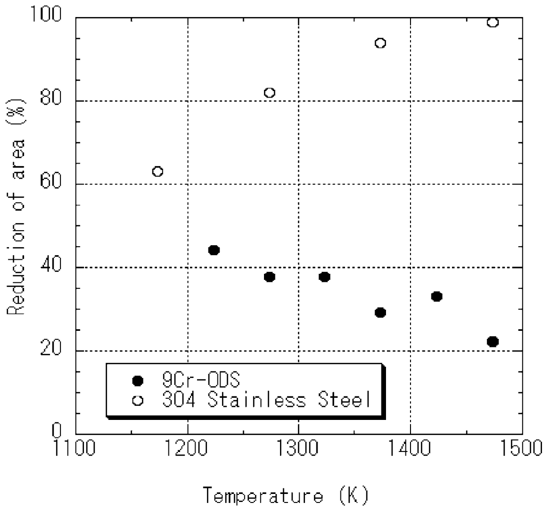


Fig. 5. Temperature dependency of reduction of area of HIPed product.

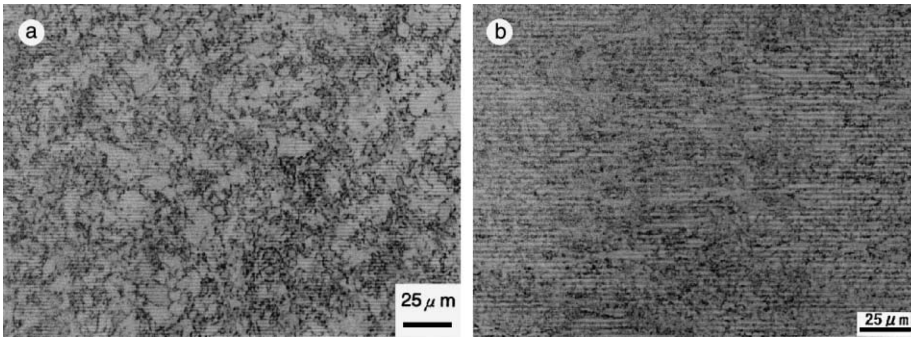


Fig. 3. Optical macrographs of (a) HIPed product and (b) annular billet made by HIP-extrusion.

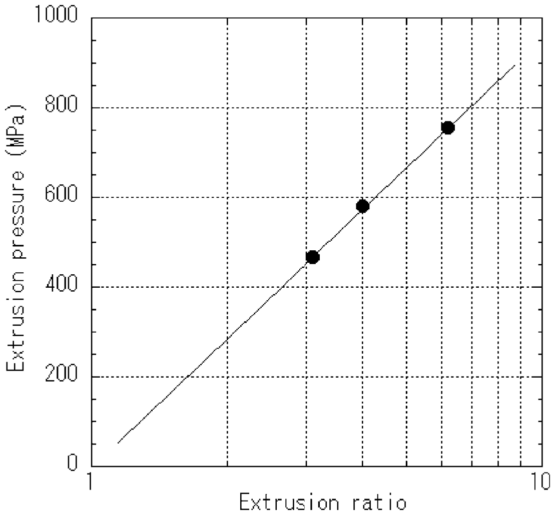


Fig. 6. Relationship between extrusion pressure and extrusion ratio for HIPed product at 1473 K.

pressure and logarithm of extrusion ratio. Thus, the extrusion pressure of HIPed product is approximately expressed in terms of deformation resistance stress, *Ym*, during hot-extrusion by Eq. (1) [5].

$$Pm = Ym \times \ln R, \tag{1}$$

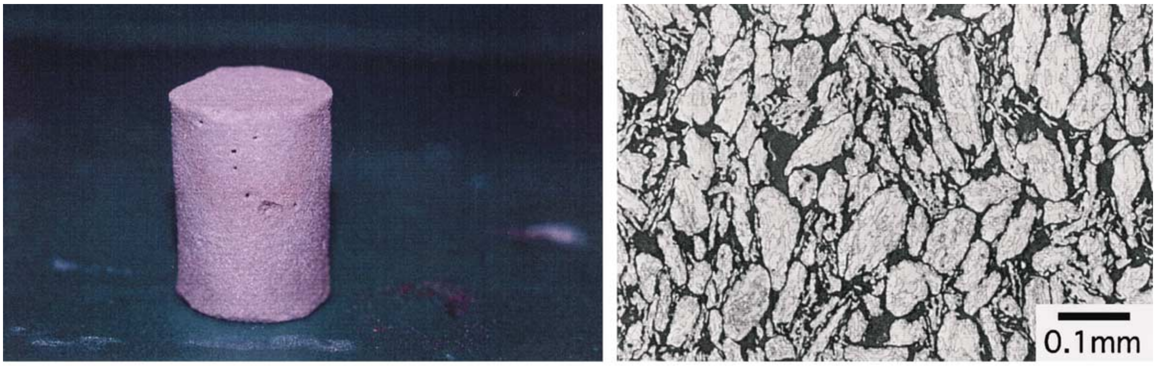
where *Pm* is the extrusion pressure and *R* is the extrusion ratio. Applying Eq. (1) to the results of present extrusion experiment, the deformation resistance stress is turned out to be about 410 MPa for this extrusion conditions, which is a sufficiently high level for plastic deformation, comparing tensile strength of HIPed product in Fig. 4. The microstructure of the hot-extruded billet of HIPed product exhibited a slightly deformed structure along the extrusion direction and thereby disappearance of pores, as shown in Fig. 3.

In addition, based on these results of small press experiments, the extrusion experiment by 2000 tons press was carried out. A HIPed product with 147 mm in outer and 32 mm inner diameters and 260 mm in length was successfully hot-extruded into an annular billet of 63.3 mm outer and 28 mm inner diameters, using a mandrel of 28 mm and die of 63.3 mm diameters. The extrusion ratio was 6.4. The deformation resistance stress was estimated to be 265 MPa in this extrusion condition. Comparing the hollow capsule extrusion experiment described in the previous section, hot-extrusion of HIPed product cannot produce the same dimension of annular billet as by the hollow capsule experiment due to limitation of the extrusion ratio. Hence, annular billet produced by HIP-extrusion requires one more time of cold-rolling to make cladding with the same dimension, comparing with billet produced by hollow capsule extrusion. It is considered, however, that HIP-extrusion should be economical process from the practical viewpoint.

4. CIP-extrusion process

A feasibility study of cold isostatic pressing (CIP) for consolidation of MM powders instead of HIP was carried out. This method uses a rubber for consolidation of MM powders at room temperature, so that no fabrication of a capsule and its removal from billet after hot-extrusion is necessary. As a result, CIP-extrusion process might be advantageous with respect to cost reduction. To evaluate the capability of CIP-products for hot-extrusion, properties of CIP-products were examined using 9Cr-ODS martensitic powders (Table 1).

The CIP processing was conducted using rubber filled with MM powders with 2 mass% paraffin at an isostatic pressure of 600 MPa. Then, the compacted CIPed products were sintered at 1423 K for 2.16×10^4 s in vacuum to get higher true density. An appearance of



(a) Appearance

(b) Cross section of optical microgragh

Fig. 7. Specimen after CIP-sintering in vacuum heating (1423 K × 2.16×10^4 s).

the CIPed product and an optical micrograph are shown in Fig. 7. The MM powders were compacted in the columnar shape, but sintering occurred only in regions of direct contact between the powder particles. Many pores remained between the particles. The true density ratio of the sintered compacted product was only 70%. It is considered that removal of pores between the particles and improvement of true density ratio can be hardly attained, even if the sintering temperature increases over 1423 K. In order to go forward with hot-extrusion, a true density ratio of over 90% is usually required. Therefore, it is judged from these tests that the hot-extrusion of CIPed and sintered products to produce the billet is not feasible.

5. Conclusion

The hollow capsule extrusion, HIP-extrusion and CIP-extrusion processes to produce a large scale annular billets for mass production were studied under economical aspects. The following results were obtained:

(1) A production of annular billet by hot-extrusion of hollow capsule filled with 9Cr-ODS MM powders has been demonstrated with higher extrusion ratio of 13. This method has a capability for mass production. A disadvantage is the need for tightly welded, rather expensive capsule to maintain MM powders integrity at hot-extrusion.

(2) The HIPed product showed a high deformation resistance stress, and thus extrusion ratio was restricted to a lower value than in the case of powder extrusion. It is, however, considered that HIP-extrusion should be a practical process from the economical viewpoint.

(3) The compaction of MM powders by CIP and sintering processing under the investigated conditions is insufficient for hot-extrusion, since its true density was lower than about 70%.

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