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Journal of Nuclear Materials 307–311 (2002) 749–757
journal of nuclear materials
www.elsevier.com/locate/jnucmat

Section 8. Dispersion-strengthened alloys

Perspective of ODS alloys application in nuclear environments

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Abstract

Oxide dispersion strengthened (ODS) steels are the most promising class of materials with a potential to be used at elevated temperature under severe neutron exposure environment. Leading technology development of ODS steels has been conducted at the Japan Nuclear Cycle Development Institute (JNC) particularly emphasizing fuel cladding application for fast reactors. This paper reviews the JNC's activities on ODS steel development as 'nano-composite materials'. Martensitic 9Cr-ODS and ferritic 12Cr-ODS steels have been successfully developed; Y_2O_3 oxide particles can be controlled on a nano-scale and high-temperature properties were noticeably improved through controlling the grain boundary structure on an atomic scale. The ODS-technology development achieved in the field of fast reactors should be effectively spun off to the fusion reactor first wall and blanket structural materials to allow for safe and economical reactor design.

1. Introduction

Ferritic/martensitic steels (FMS) are a primary candidate for the advanced fast reactor cladding/duct materials as well as fusion DEMO plant first wall and blanket structural materials because of their advantage to radiation resistance up to high neutron dose as high as 200 dpa [1,2]. Their utilization is, however, limited to around 600 °C, which is due to inferior tensile and creep strength at higher temperatures. To achieve higher plant operation temperature for improved thermal efficiency, efforts have been made to improve high-temperature properties by means of controlling alloying elements and heat-treatment with stabilized carbide precipitates in FMS, especially for application in the power-generation industry [3]. Oxide dispersion strengthened (ODS) FMS are promising materials with a potential to be used at elevated temperatures due to the addition of extremely thermally stable oxide particle dispersion into the ferritic/martensitic matrix. The development of ODS FMS has been conducted in the field of fast reactor fuel cladding application [4–8] and fusion reactor materials application [3,9–12].

A leading technology development of ODS FMS has been conducted in the Japan Nuclear Cycle Development Institute (JNC) particularly emphasizing fuel cladding application for fast reactors. This technological R&D is believed to extend the performance of reduced activation ferritic steels as a system applicable in fusion structural materials. In this paper, JNC's activities on the development of ODS FMS are reviewed. The underlying guideline for the processing method in the shape of panel and pipe will be provided on the basis of comparison of the current baseline properties with the requirements from the tentative fusion reactor materials design. Future work needed for ODS development also is presented.

2. Technology development for fast reactor fuel cladding application

2.1. JNC's activity and progress

The research and development of the ODS FMS, as a prospective cladding material for the advanced fast reactor, are being conducted since 1987 in JNC. Fundamental

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0022-3115/02/$ - see front matter
PII: S0022-3115(02)01043-7

studies concerning optimization of mechanical milling (MM) processing as well as effects of alloying elements on the high-temperature mechanical strength had been carried out in cooperation with fabrication vendors [13,14]. Based on the results of those studies, the manufacturing of thin-walled cladding had been tried with hot-extrusion and warm-rolling processes in 1990 [5]. This initial effort revealed that the manufactured claddings had not only degraded creep rupture strength in bi-axial hoop direction in comparison with longitudinal uni-axial direction, which is so-called strength anisotropy, but also significantly poorer ductility in the hoop direction. Based on the fundamental study collaborated with Yoshinaga's group of Kyusyu University, these unexpected mechanical properties of the manufactured ODS claddings were attributed to the grain boundary sliding among grains extremely elongated parallel to the rolling direction [15].

In order to make equi-axed and homogeneous grains, two kinds of approach had been experimentally explored using the extruded bars up to 1994: α to γ phase transformation for martensitic 9Cr-ODS steels especially aiming at radiation resistant alloys and on the other side recrystallization processing for ferritic 12Cr-ODS steels aiming at corrosion resistant alloys [16–18]. From 1995 up to 2000, an extensive technological breakthrough has been accomplished for manufacturing thin-walled claddings to prevent crack initiation at an intermediate manufacturing process and for assuring both superior internal creep strength and ductility with homogeneous grain morphology on the basis of phase transformation and recrystallization processing [19–22]. The detail of this technological development will be described in the following sections. Currently, the materials development itself has been almost accomplished by realizing cold-rolling cladding manufacturing and assuring high temperature strength and ductility toward the target required from the design study of commercialized fast reactors. The main study is gradually shifted to the material system engineering phase, e.g. mass production processing, joining, inspection, irradiation and engineering data-base, to assure ODS steels of their applicability as a fast reactor fuel pin system [23].

2.2. Nano-scale oxide particle control

The characteristic feature of ODS steels is to introduce the Y_2O_3 oxide particles into the matrix, which serve as a block for mobile dislocations to improve the high-temperature strength and as a sink of point defects induced by radiation displacement to maintain superior radiation resistance [24].

The effects of co-addition of elements such as Ti, Nb, V, Zr in ferritic 12Cr-ODS steels were extensively investigated; size distribution of the oxide particles by image-analyzing of transmission electron micrographs are shown in Fig. 1 [13]. In case of simple addition of

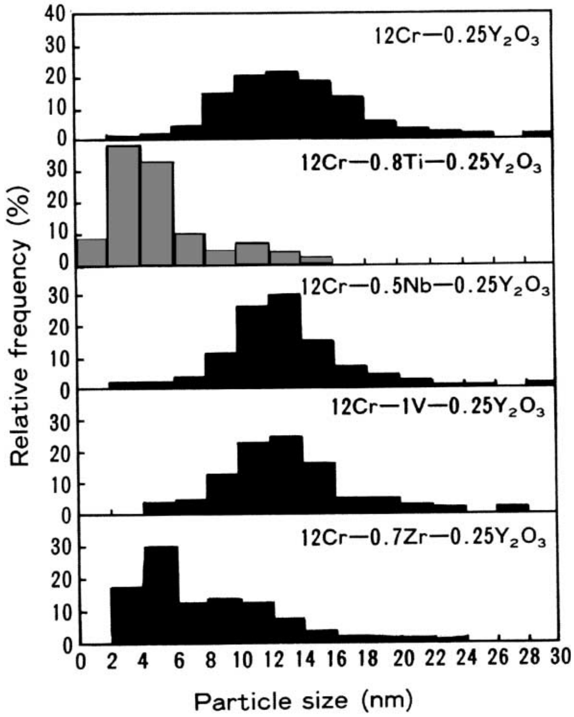

Fig. 1. Size distribution of oxide particles determined by TEM in 12Cr-ODS ferritic steels in various elements addition: Ti, Nb, V and Zr [13].

Y_2O_3 in ferritic steel, stable oxides can be detected with the size of more than 10 nm, which is similar in size to the original 20 nm of added Y_2O_3 particles. An addition of a small amount of Ti element sufficiently reduces the oxide particle to around 3 nm size. This surprising effect is concomitantly supported by a significant improvement of the creep rupture strength by addition of Ti in ferritic 12Cr-ODS steels. The effectiveness of Zr to reduce the oxide particle size is weaker than that of Ti, which was accompanied by less improvement of creep rupture strength.

The formation mechanism of extremely fine oxide particle induced by addition of Ti alone was studied by the co-authors using X-ray diffraction analyses of the mechanically milled powders during heating up to 1300 °C from room temperature. The results of experiment are shown in Fig. 2 [25]. As-mechanically milled powders do not give the specific diffraction peak. After annealing at elevated temperatures above 1100 °C, however, diffraction peaks appear corresponding to titania, complex Y–Ti–O compounds and yttria. A similar result is reported by Takaki and co-workers [26]. Therefore, it is considered that thermally stable Y_2O_3 decomposed into yttrium and oxygen atoms, and dissolved into the 12Cr ferritic steels at the process of MM during introduction of super-heavy energy. Followed by annealing above

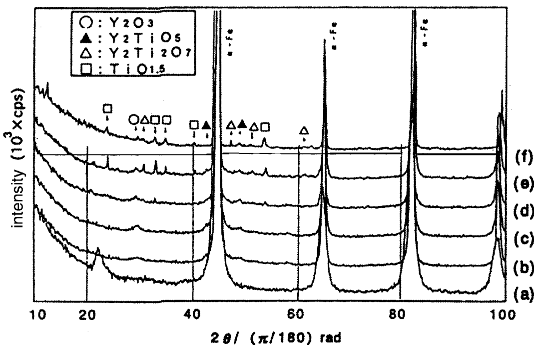

Fig. 2. Results of X-ray diffraction analyses on mechanically milled powders [25]: (a) as-mechanically milled, (b) after annealing at 900 °C for 1 hr, (c) after annealing at 1000 °C for 1 hr, (d) after annealing at 1100 °C for 1 hr, (e) after annealing at 1200 °C for 1 hr and (f) after annealing at 1300 °C for 1 hr.

1100 °C, dissolved elements would be bonded with Y, Ti and oxygen, and extremely fine Y–Ti–O complex oxides would precipitate as more stable oxide particles. It is to be noticed that ODS steels should be challenging nano-scale composite materials that can be controlled through dissolution and precipitation of oxide particles by high energy MM followed by heat-treatment.

2.3. Grain structure control

A cold-rolling process to manufacture claddings leads to the extremely elongated fine grain morphology parallel to the rolling direction, which induces higher creep rupture strength in this direction due to the large grain aspect ratio [27]. The transmission electron micrograph of these finely elongated grains is represented in Fig. 3. However, a primary stress mode in the cladding of the fission gas pressurized fuel pins is the transverse hoop direction, where ductility loss and deterioration of creep rupture strength are taken place. Thus, making equi-axed grains is a key technology for realizing the ODS steel cladding.

As an approach to overcome this hurdle, technology to induce the α to γ phase transformation in martensitic 9Cr-ODS steels was developed [22]. The characteristic Ac_1 and Ac_3 points of 9Cr–0.13C–2W–0.2Ti–0.35Y_2O_3 (mass%), that are the temperature at which the transformation of ferrite–martensite (α) into austenite (γ) is starting and finishing respectively, are 884 and 960 °C at a heating rate of 8.3×10^{-2} °C/s. This higher Ac_1 point of the martensitic 9Cr-ODS steel should be advantageous for fuel pin applications, especially when the cladding is rapidly heated up at the event of loss of coolant flow in the fast reactors. Fig. 3 shows the TEM microstructure of the martensitic 9Cr-ODS cladding. It has a typical tempered martensitic structure with a lath size of less than 1 μm. This fine and homogeneous grains are formed in the prior austenite grains that are finer due to grain growth retardation by existence of Y_2O_3 particles at the normalizing condition.

Another approach to destroy the elongated grains in as-rolled claddings is to apply the recrystallization processing for ferritic 12Cr-ODS steels with a composition of 12Cr–0.03C–2W–0.3Ti–0.23Y_2O_3 (mass%) [21]. The recrystallization diagram was developed in the field of yttria-excessive oxygen contents [16]. The yttria content strongly affects the extent of recrystallization, and must be restricted to a content less than 0.25 mass%. Fig. 3 also represents the TEM micrograph showing recrystallized large grains. Concomitantly, texture change is an important parameter for recrystallization. The cold-rolling gives rise to a typical texture of {1 1 1} ⟨1 1 0⟩ parallel to the rolling direction, and recrystallization induces more randomness of this texture [15]. This recrystallized grain structure caused improvement in the high-temperature strength and ductility of the hoop direction.

2.4. Tube manufacturing

The ODS steels are too hard to manufacture cladding by the cold-rolling process alone. Based on the extensive

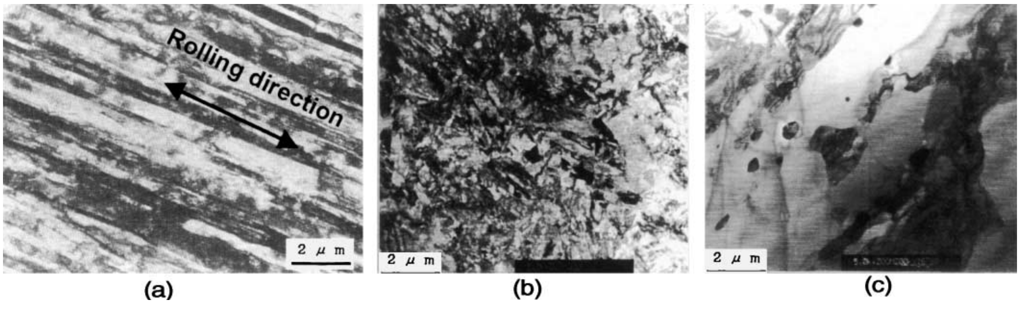

Fig. 3. TEM micrographs of thin foils in cold-rolled cladding (a), tempered martensitic structure of 9Cr-ODS (b) and recrystallized structure of 12Cr-ODS (c).

investigation of the continuous cooling transformation diagram of 9Cr–0.13C–2W–0.2Ti–0.35Y_2O_3, a less than critical cooling rate of about 150 °C/h gives rise to the ferrite phase at room temperature without martensite, which induces lower hardness. Applying this heat treatment technique, cold-rolling by pilger milling was conducted under the softened ferrite structure [28]. The solid circles in Fig. 4 show the hardness change of martensitic 9Cr-ODS steel in the process of cladding manufacturing by repeating cold-rolling and heat treatment. Each pass introduces approximately 50% cold-rolling and leads to the final dimension of 8.5 mm diameter and 0.5 mm wall-thickness. The hardened cladding due to accumulation of cold-rolled deformation can be successfully softened by the heat treatment with a slow cooling rate of 150 °C/h after normalizing at 1050 °C. The final heat-treatment consists of normalizing at 1050 °C for 1 h, followed by tempering at 750 °C for 30 min.

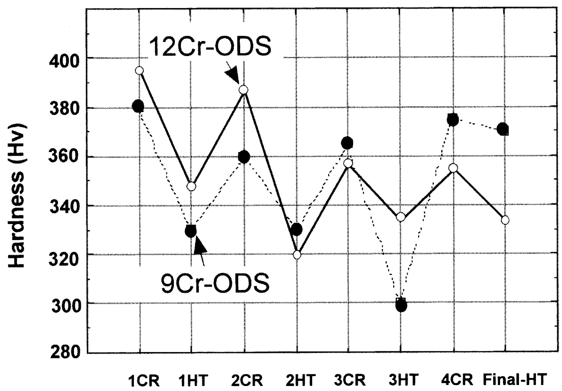

Fig. 4. Hardness change in the process of cold-rolling cladding manufacturing in martensitic 9Cr-ODS and ferritic 12Cr-ODS steels.

The hardness change of ferritic 12Cr-ODS steels is shown by the open circles in Fig. 4. The intermediate heat-treatment was conducted by annealing at 1100 °C and finally at 1150 °C to yield the recrystallized grain structure [21].

2.5. High-temperature strength characterization

The tensile properties of the manufactured martensitic 9Cr-ODS cladding are represented in Fig. 5 for the hoop stress mode conducted by ring tests at room temperature to 800 °C [22]. The martensitic 9Cr-ODS cladding showed the superior ultimate tensile strength (UTS) and uniform elongation (UE) as well over the entire temperature when compared with the conventional ferritic–martensitic stainless steel, PNC-FMS [1]. The ratios of UTS and UE to those of PNC-FMS are also represented in Fig. 5. From these comparisons, the strength and ductility improvement in the martensitic 9Cr-ODS claddings are prominent especially above temperatures around 600 °C; this advantage could arise from the retardation of recovery and continuing work-hardening due to pinning the dislocations by finely distributed oxide particles.

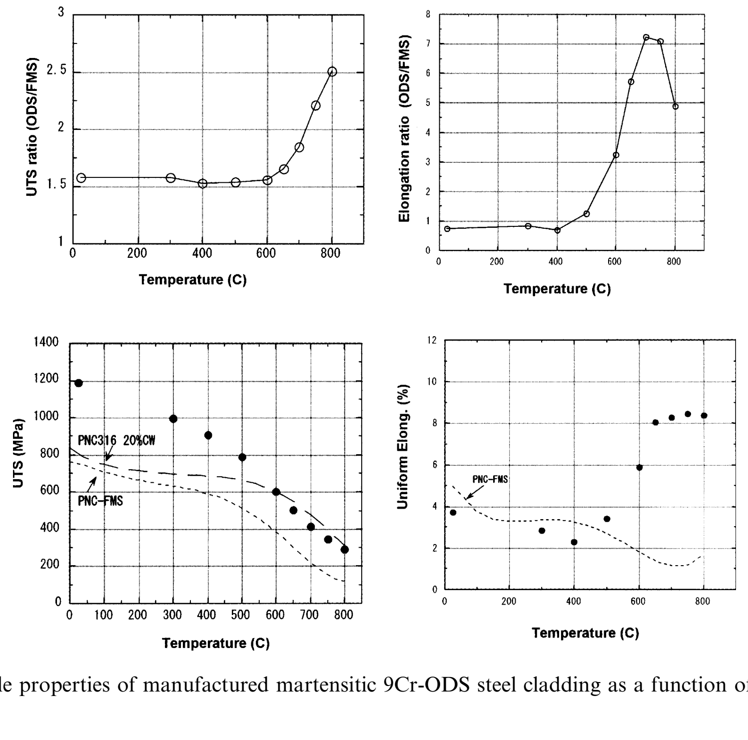

Fig. 5. Tensile properties of manufactured martensitic 9Cr-ODS steel cladding as a function of temperature.

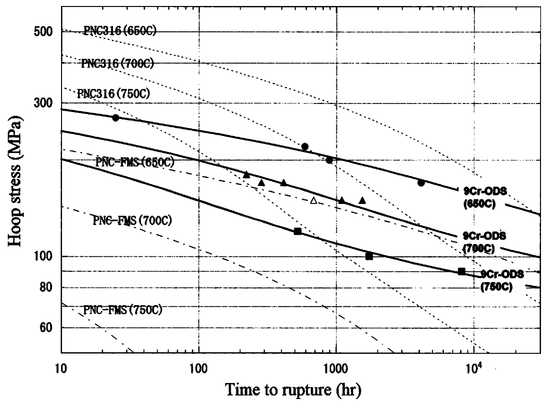

Fig. 6. Creep rupture properties of manufactured martensitic 9Cr-ODS steel cladding, comparing with those of PNC-FMS and PNC316.

The creep rupture strength of the manufactured martensitic 9Cr-ODS cladding at 650, 700 and 750 °C is shown in Fig. 6 in comparison with those of PNC-FMS and modified 316 SS with 0.1 mass% Ti and 0.1 mass% Nb (PNC316) [22]. These curves were predicted, based on the Larson–Miller parameter method. We must emphasize that the strength anisotropy perfectly disappears in the hoop and longitudinal directions, and the internal creep strength level approaches the target of 120 MPa for 10 000 h at 700 °C that is required from the advanced fast reactor fuel design. This strength level is far beyond that of PNC-FMS, and superior to even PNC316 beyond 1000 h at 750 °C.

This noticeably improved strength level in the martensitic ODS claddings is owing to the extremely fine distribution of oxide particles that was revealed via transmission electron micrographs: 3 and 40 nm for an average diameter of oxide particle and surface–surface average distance between particles, respectively. Theoretical analysis of the threshold stress for deformation using the above dispersion parameters suggested that a pure strength level determined by the oxide particles-dislocation interaction should be lying above the measured strength level; ODS steel cladding still has a potential to allow a furthermore increase in the creep rupture strength. Making a coarser grain formation would be a unique solution to approach the dispersion strength level on the basis of findings that accelerated deformation could arise from a grain-boundary sliding among extremely fine and homogeneous grains less than 1 μm in martensitic 9Cr-ODS claddings as shown in Fig. 3 [29].

2.6. Joining technology development

Joining technology between ODS steel cladding and the end-plugs has been developed to apply for the fast reactor fuel pin fabrication at Tokai-work of JNC. A pressurized resistance welding (PRW) method is utilized as solid state joining [30]. The method is based on electrical resistance heating of the components while maintaining a continuous force sufficient to forge weld without melting of the components. The contact force between cladding and end-plug is performed by a pneumatic-hydraulic system. The welding parameters, e.g. electric current, voltage and contact force, are controlled by a programmable system. The typical conditions are a 600 kgf contact load with 16 kA current for 16 ms after holding for pre-heating by 6 kA current. Fig. 7 represents the cross section of the joining parts, where ODS steel is also used in the end-plug. The forged bond line is identified in this photograph. The PRW method produces metallurgically bonded welds, keeping the original dispersoid distribution and ODS fine grain structure unchanged. Post-weld machining was performed to true the end-plug diameter and remove the external upset material. The tensile and internal creep rupture tests were conducted at room temperature to 800 °C; it was demonstrated that rupture occurred at the cladding tube itself keeping integrity of bonded parts. Upgrade of the joining process qualification is continued. In addition, an ultrasonic testing method has been developed to assure that non-destructive integrity of the joining between cladding and end-plug.

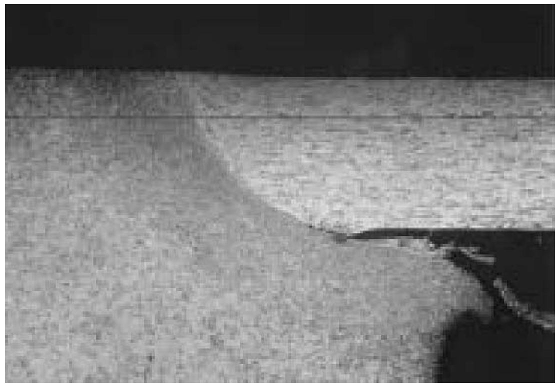

Fig. 7. Optical micrograph of the cross section in the PRW joining part between ODS steel cladding and ODS steel plug.

2.7. Radiation resistance

The irradiation performance of ODS steels is investigated by means of neutron, ion and electron irradiation experiments. The ring tensile specimens of martensitic 9Cr-ODS and ferritic 12Cr-ODS steels have been irradiated in the Japanese experimental fast reactor JOYO up to 15 dpa and at a temperature range of 400–550 °C. It was confirmed that within these irradiation conditions strength and ductility levels are adequately maintained. From the TEM observation, a significant stability of oxide particles and a dislocation density in ferritic ODS steels have been confirmed under neutron irradiation [31]. Higher neutron dose data will be acquired in future.

In parallel, the high-voltage electron irradiation experiment has been conducted at Hokkaido University; the effects of the MM atmosphere, e.g. argon and helium, and the tempered martensite phase on the void nucleation and growth were clarified [32,33]. The helium release behavior from ODS steels also has been investigated at Kyoto University in comparison with conventional FMS.

3. Technology assessment for fusion reactor materials

3.1. Target mechanical properties

In the first wall and breeder blanket structural materials for the DEMO fusion reactor, the tentative design requires an UTS of 500–550 MPa at 650 °C after 15 MW/m^2 to maintain their integrity for the thermal stress, when reduced-activation ODS ferritic steels are applied [34]. Fig. 8 compares the tensile strength behavior of as-manufactured F82H (8Cr–0.1C–2W–0.2V–0.04Ta) [3] and martensitic 9Cr-ODS steel tubes. The design requirement for the tensile strength at 650 °C are just located at the strength lines of martensitic 9Cr-ODS steels. It is recommended to add yttria more than 0.35 mass% as a typical fast reactor cladding to further improve and adequately satisfy the design requirement, since panel-type forming would be easier than manufacturing of fast reactor thin-walled cladding.

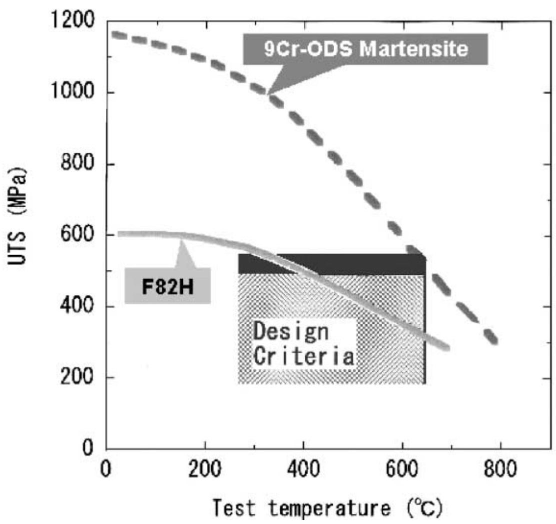

Fig. 8. Comparison of UTS in martensitic 9Cr-ODS steel cladding, F82H and tentative design criteria for fusion reactor materials.

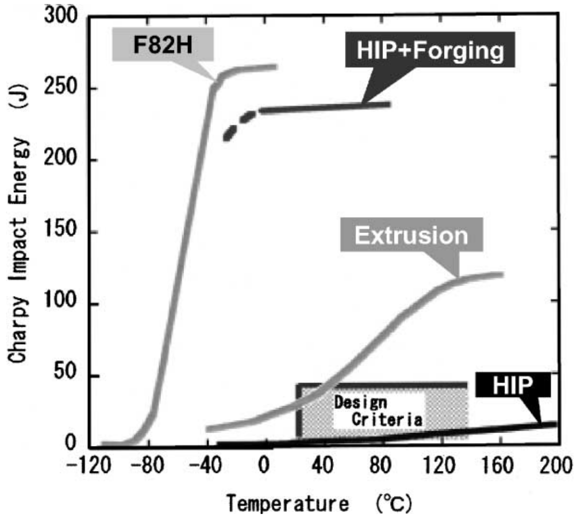

Fig. 9. Comparison of Charpy impact energy in various type of manufacturing for ferritic 13Cr-ODS steels, F82H and tentative design criteria for fusion reactor materials (full size dimension).

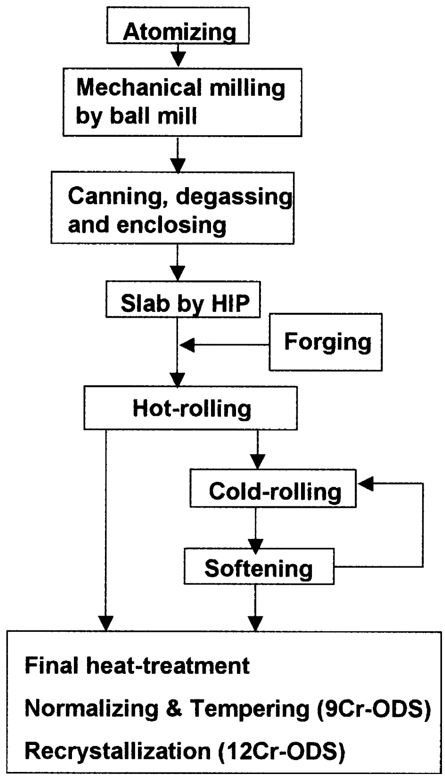

Fig. 10. Proposed production process for fusion first wall panel.

Another specific aspect for reduced activation ferritic/martensitic steels for fusion application as structural materials is the concern with the Charpy impact property [35]. The measured Charpy impact energy of the specimens formed by various processing methods is shown in Fig. 9 with a tentative design requirement for the DEMO fusion reactor [34]. The absorbed energy of hot-extruded usual bar with a chemical composition of 13Cr–0.02C–3W–0.5Ti–0.4Y_2O_3 ferritic steel lies around the design criteria. These specimens were prepared in parallel to the extruded axis. The hot isostatic pressing (HIP) specimens lead to the extremely low level of absorbed energy that could be attributed to the existence of pores at the boundaries of sintered particles. However, HIP followed by forging processing raises the absorbed energy significantly and approaches that of F82H. This improvement could be attributed to the disappearance of pores at sintered particle boundaries by accumulation of plastic deformation. As clearly indicated that the Charpy impact property strongly depends on the forming process, it should be paid attention to utilize the near-shape forming by HIP processing alone.

3.2. Production processing

The production processes of a thick-panel and large-diameter pipe of ODS steels must be established to apply them to the heavy sections of future fusion first wall and blanket systems. The developed technology for manufacturing the fast reactor fuel cladding should be directly applicable to the production process of fusion reactor pipe as described in Section 2.4. In the case of panel production, however, the process development for this particular product should be required.

Fig. 10 shows the proposed panel production process for fusion first wall application. A slab should be manufactured by HIP from the mechanical milled powders, and then a large-scale size panel can be directly produced by means of hot-rolling. The hot-rolling process should be necessary in the course of the production process, since the HIP products yield a uniform structure but their Charpy impact properties are degraded as described above. As an alternative processing route, cold-rolling and subsequent heat-treatment are repeated to make the final panel in about 2 m $\times$ 2 m size and desired thickness with sufficiently dimensional accuracy. It is inevitable to soften the hardened cold-rolled panel by means of furnace cooling for martensitic 9Cr-ODS steels and recrystallization-annealing for ferritic 12Cr-ODS steels. The direct production of the final shape by hot-rolling would be applicable under certain circumstances. At the final stage, a heat-treatment to make equi-axed grains is necessary: α to γ phase transformation for 9Cr-ODS steels and recrystallization processing

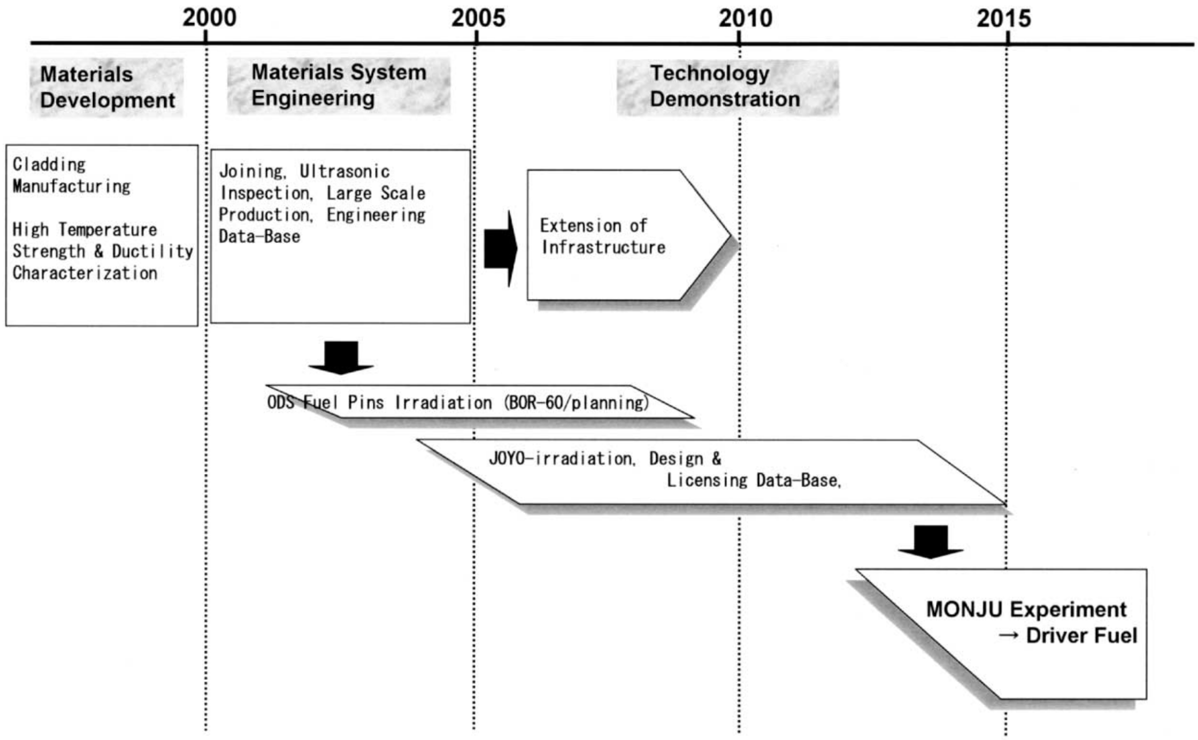

Fig. 11. Future work and schedule for demonstrating ODS fuel pins in the advance fast reactors.

for 12Cr-ODS steels. The large-scale equipment capable for production processing already exists in the steel industry.

4. Future work

The future work toward realizing ODS fuel pins for advanced fast reactors is schematically represented in Fig. 11. We are currently starting the stage of materials system engineering, where cladding manufacturing dimension accuracy, joining with plug, inspection by ultrasonic inspection, large scale production and construction of engineering data-base are included. For technology demonstration as a fuel pin system, irradiation tests of ODS fuel pins, that are under preparation, will be conducted using the Russian experimental fast reactor BOR-60 and JOYO. Through accumulation of an abundant irradiation data-base for design and licensing, the final target should be to apply ODS fuel pins as driver fuels of the prototype fast breeder reactor MONJU. These activities should be timely reflected to the fusion ODS development.

5. Conclusion

The martensitic 9Cr-ODS and ferritic 12Cr-ODS steels have been successfully developed as promising fuel cladding materials in fast reactors. Essentially in the ODS steels, the distribution of stable Y_2O_3 oxide particles can be controlled on a nano-scale that serves as a strong block for mobile dislocations and as a sink for radiation defects at the particle-matrix interfaces. Furthermore, high-temperature strength and ductility are far advanced through controlling the grain boundary structure. Thus, the ODS steels should be challenging 'nano-composite materials' to be applicable to nuclear materials. The appropriate processing method on a large scale including MM of ODS powders and consolidation by HIP and/or hot-extrusion was proposed for the industrial application of fusion reactor materials. The ODS-technology development achieved in the fast reactor fuel claddings should be effectively spun off to the fusion reactor first wall and blanket structural materials to allow for safe and economical reactor design with an enlarged design window.

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