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Structural materials by powder HIP for fusion reactors

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Abstract

Tokamak blankets have complex shapes and geometries with double curvature and embedded cooling channels. Usual manufacturing techniques such as forging, bending and welding generate very complex fabrication routes. Hot Isostatic Pressing (HIP) is a versatile and flexible fabrication technique that has a broad range of commercial applications. Powder HIP appears to be one of the most suitable techniques for the manufacturing of such complex shape components as fusion reactor modules. During the HIP cycle, consolidation of the powder is made and porosity in the material disappears. This involves a variation of 30% in volume of the component. These deformations are not isotropic due to temperature gradients in the part and the stiffness of the canister. This paper discusses the following points:

(i) Availability of manufacturing process by powder HIP of 316LN stainless steel (ITER modules) and F82H martensitic steel (ITER Test Module and DEMO blanket) with properties equivalent to the forged one.
(ii) Availability of powerful modelling techniques to simulate the densification of powder during the HIP cycle, and to control the deformation of components during consolidation by improving the canister design.
(iii) Material data base needed for simulation of the HIP process, and the optimisation of canister geometry.
(iv) Irradiation behaviour on powder HIP materials from preliminary results.

1. Introduction

Tokamak blankets have very complex shapes and geometries with double curvatures and embedded cooling channels. The well-known manufacturing techniques such as forging, machining, casting and welding generate very complex fabrication routes. In some cases, homogeneity of the material cannot be assured by such processes. Powder HIP appears to be one of the most suitable techniques for the manufacturing of such complex shape components as fusion reactor modules. The application of this process to structural material for fusion reactors is described, mainly 316LN stainless steel for the ITER blanket, and F82H martensitic steel for the ITER Test Module and DEMO. Behaviour of these materials under neutron irradiation is also discussed.

2. Hot isostatic pressing process

Hot Isostatic Pressing (HIP) is a versatile and flexible fabrication technique that has a broad range of commercial applications. HIP combines elevated temperature (up to 2000°C) and gas pressure (up to 200 MPa) to consolidate powder and produce parts. Powders used for these applications are obtained by gas atomisation or rotating electrode pulverisation. The shape of the powder particle is nearly spherical, and a large grain size distribution is obtained. This powder is screened before use, and typical grain size distribution used is between 45 and 500 μm, depending on the alloy and the atomisation process.

To be consolidated, the powder is encapsulated in a canister. This canister is filled with the powder, degassed for several hours at medium temperature (200°C), and closed. Filling is done under vibration to obtain a uniform distribution of the particles. A relative density is defined as the ratio of the apparent density of the material to the density of the fully dense material (with no

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porosity). The initial relative density obtained after filling is called the tap density and is dependent on the material. For gas atomised powders, this tap density is between 60% and 70%.

The canister is put into the HIP vessel, and pressure is applied to the canister by a gas. Pressure and temperature are raised simultaneously, then maintained for some hours at the specified values, and finally decreased. For stainless steel material 316LN, reference material for ITER blanket, typical parameters are 4 h to increase temperature and pressure to 1125°C and 120 MPa respectively, then 10 h dwell time, and finally 4 h to decrease temperature and pressure [1]. This is shown in Fig. 1. In the case of F82H martensitic steel, temperature and pressure are increased in 1 h to 800°C and 100 MPa, then increased in 30 min to 1050°C and 140 MPa. After holding for 2 h, the temperature is decreased very rapidly at a rate higher than 10°C/min from 1050°C to 500°C (Fig. 2). This rapid cooling is called HIP quenching. After HIP, the material is fully dense with no porosity. This involves large deformations of the canister, up to 35% in volume. Due to canister stiffness and temperature gradients in the powder, the deformations are not isotropic. To define the initial shape of the canister, modelling must be used for complex shapes.

Experiments have been performed using the CEREM laboratory HIP vessel. Its dimensions are 100 mm in diameter and 200 mm in height. This laboratory HIP furnace is shown in Fig. 3. Industrial equipment dimensions are up to 1500 mm in diameter and 3000 mm in height. Concerning HIP quenching, new HIP vessels are capable of cooling rates up to 50°C/min for a load of nearly one ton of material.

3. PRECAD®

A special tool has been developed by CEA/CEREM for the design and modelling of canisters used to produce net-shape parts by HIP. This tool is constituted of three modules: PreCAD/D for the design of the parts and meshing, PreCAD/M for the modelling of consolidation of the powder, and PreCAD/B for the data base materials including powders and container. A general description of this tool is given in Fig. 4.

3.1. Computer assisted design module PRECAD/D

PRECAD/D is the computer assisted design (CAD) module. It is used to design both the part to be realised and the tools necessary to produce this part from powder without subsequent machining. The geometry of the design part can be provided directly by the end-user in the form of a data file, using usual CAD interfaces, such as IGES. Each part (tool, powder, punches, canister, …) are automatically meshed in this CAD module. Meshes, material characteristics, and boundary conditions are then sent to the modelling module.

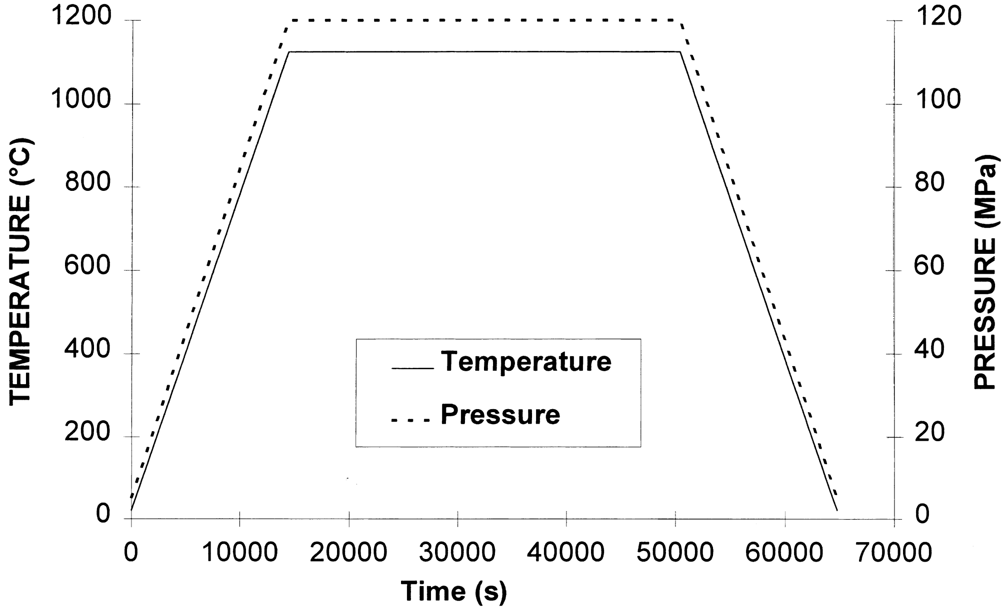

Fig. 1. HIP cycle for consolidation of 316LN stainless steel powder.

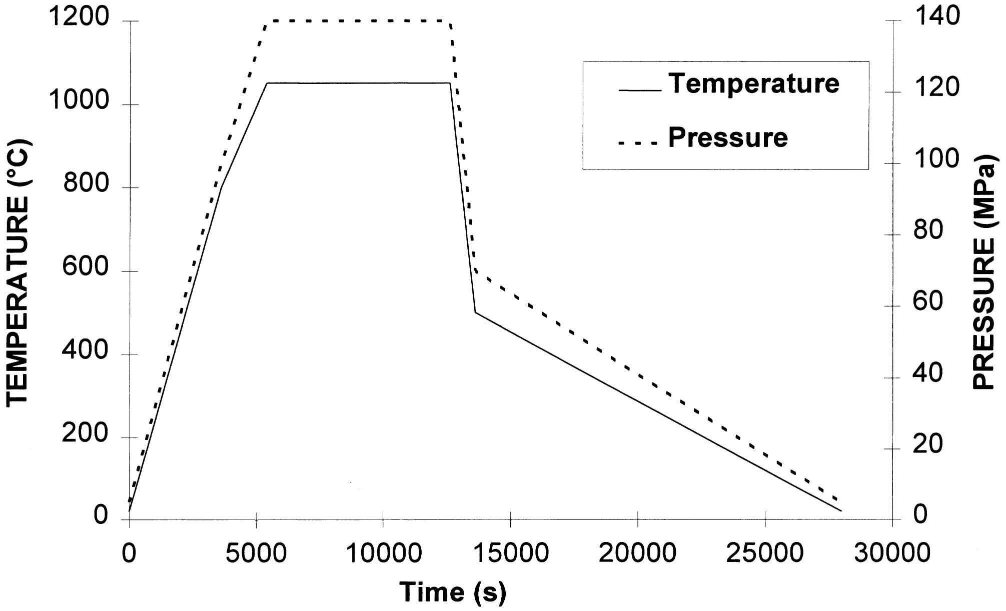

Fig. 2. HIP cycle for consolidation of F82H martensitic steel powder.

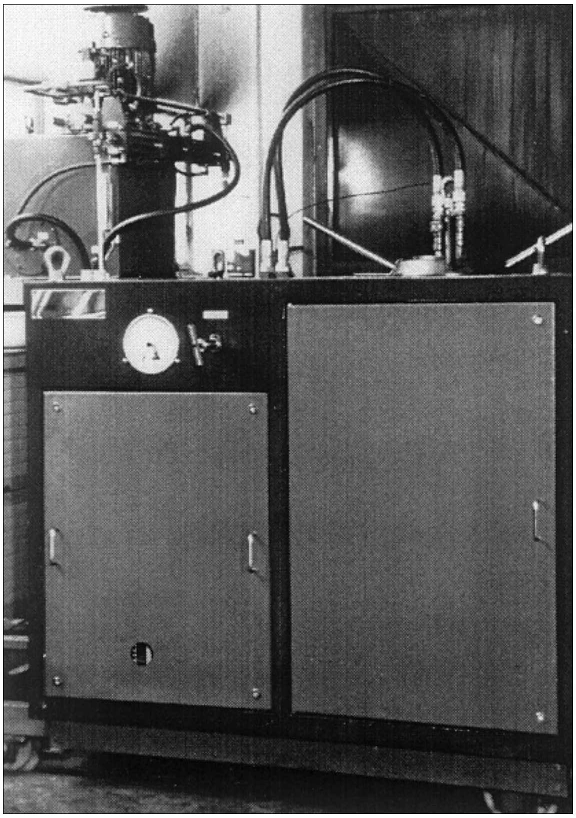

Fig. 3. Laboratory HIP furnace of CEA/CEREM.

3.2. Modelling module PRECAD/M

To simulate the consolidation of powder by HIP, a macromechanical approach is used. Powder is considered as a continuous medium with relative density as a state variable. The relative density is defined as the ratio of the apparent density to the density and fully dense material.

The finite element method (FEM) is used in PRECAD/M. Simulation is available for plane strain, plane stress, generalised plain strain, axisymmetric and tridimensional analysis. The modelling procedure enables the user to carry out an incremental thermo-mechanical non-linear analysis. For each time step, a thermal procedure first calculates the temperature map on the mesh. The procedure allows linear and non-linear computations with conduction, convection and radiation. The thermo-dependant material parameters are then calculated and sent to the mechanical non-linear procedure. There, the non-linearity can result either from the material (visco-plasticity), or from large displacements, even from both. The thermal history of the material is modelled, giving the possibility to predict the mechanical properties as well as the microstructure of the consolidated powder at the end of the process.

3.3. Data base module PRECAD/B

Simulation requires the constitution of material data files, containing physical and thermo-mechanical

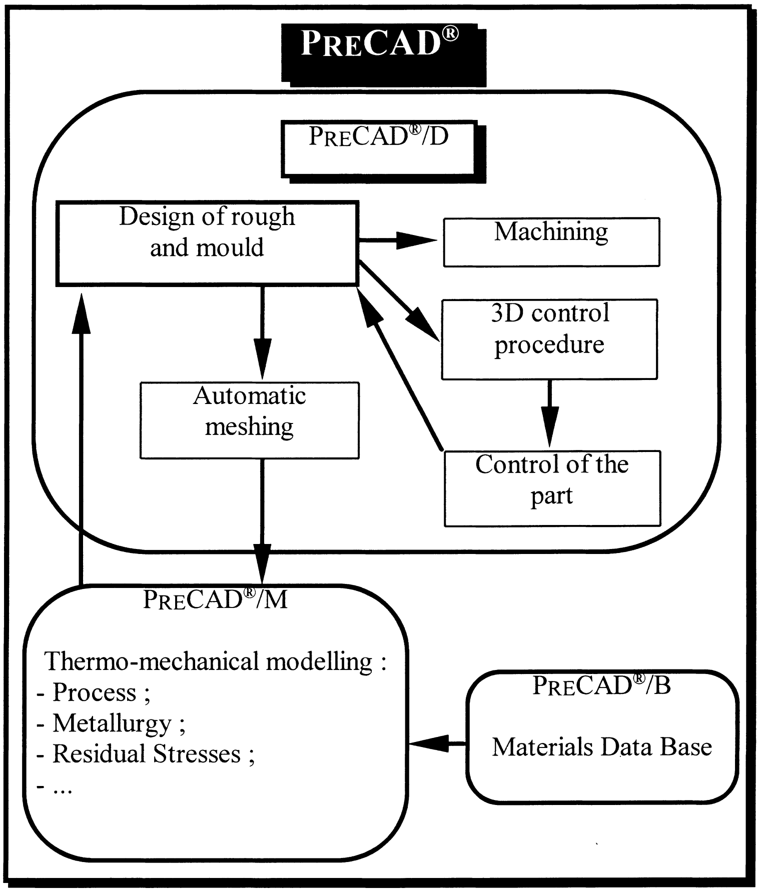

Fig. 4. General description of PreCAD® tool and its modules.

properties of the materials (Young modulus, poisson's ratio, thermal expansion coefficient, creep properties, thermal conductivity,…) including coefficients of the porous constitutive equations for the powder.

These data are determined experimentally. In the case of HIP, the model of Abouaf [2] is used. The material parameters are four functions, creep parameters A and n that depend on temperature, and two rheological functions f and c depending on the relative density only. The way of determining these parameters is presented in details in [3]. Data have been determined for an ITER grade 316LN stainless steel [4] and is ongoing for F82H martensitic steel, a possible material for the ITER Test Module and DEMO blanket.

4. Applications

PRECAD® is used to optimise canisters for producing net-shape parts by HIP. First the design of the part to be manufactured is provided by the end user. An initial design of the tools and canister is made, and modelling gives the final geometry of this design. This final shape is compared to the desired one, allowing iterative work on the design of the tool to optimise this geometry. It is also used to shorten the time step at high temperature and pressure, in order to reduce the cost of hipping. This is possible because the density map in the powder is known at each time step. The accuracy of the prediction of the simulation has been found to be equal to ±50 μm on a demonstrator of 100 mm diameter and 70 mm height [5]. Prediction of the relative density is better than 1% [3] for HIP applications on TA6V powders.

4.1. Validation process

Validation of the simulation is performed by defining a small scale mock-up. The canister is realised, and each component is measured before assembling. The initial relative density is calculated by measuring the volume of the canister and the mass of powder put into it. The filled canister is measured just before hipping. The HIP cycle is then run, and the deformed canister is again measured. When it is possible, the part is also measured after decanning, before final machining. Modelling is performed using the real initial data (measured geometries of canister and tools, calculated initial relative density, real HIP cycle). Results from the modelling and the manufacturing are then compared, to estimate the accuracy of modelling.

4.2. Manufacturing of a validation part

In the case of 316LN ITER grade stainless steel powder, a block has been manufactured [6]. A description of the canister is given in Fig. 5, and dimensions are given in Table 1. The canister material is 304L stainless steel, made from welded sheets and tubes of 1.5 mm thick. It has been produced by Crucible Research, United States, in the scope of an International Collaborative Programme on Modelling of Metal Powder Forming Processes [7]. The initial relative density is 69%, and the HIP cycle used to manufactured the part is the one shown in Fig. 1, at 1125°C and 120 MPa for 10 h. The part is fully dense at the end of HIP cycle. Final dimensions are also given in Table 1.

4.3. Modelling of validation part

The part presents a plane of symmetry (Plane A-B), so one half of the part is considered. A mesh is generated in PRECAD/D, and 10 node quadratic tetrahedral elements are used in that case. A temperature gradient of 10°C maximum occurs in the part during the temperature increase, as shown in Fig. 6. At the end of HIP cycle, the part is calculated to be fully dense. In fact, it is shown that for such a mock-up, full density is reached before the temperature and pressure step. The final modelled shape is cut by a plane defined by the axes of the two holes of the part and is presented in Fig. 7. Some 3D effects are visible on this figure. The tube is larger in the middle plane after hipping. This is due to canister stiffness. To fully consolidate the powder, some deformations of the tubes are necessary. This effect is emphasised because of the welds performed to link tube and plate. Defining an error by the ratio of simulated displacement to measured displacement, we obtain an accuracy between simulation and measurement better than 10%. To compare, if one assume an isotropic deformation of the canister, the error is then 40%. These results are given in Table 2.

Table 1
Dimensions of the validation part for 316LN consolidation by HIP

Dimension	Initial value before HIP (in mm)	Final value after HIP (in mm)
A	160.807	143.561
B	230.990	206.324
C	39.472	34.442
D	21.184	19.939
E	72.923	64.440
F	68.351	60.960
G	35.103	33.198
H	38.075	34.112
I	76.987	68.250

The reference cycle has been defined to assure the full consolidation on large components such as the ITER blanket [1]. On a small scale mock-up such as the validation part, a shorter time step for the temperature and pressure cycle can be used. The modelling tool is found

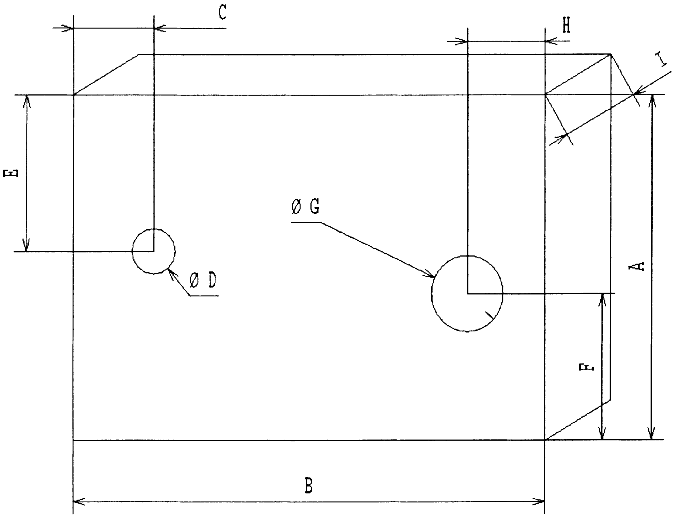

Fig. 5. Validation part for modelling of 316LN consolidation by HIP.

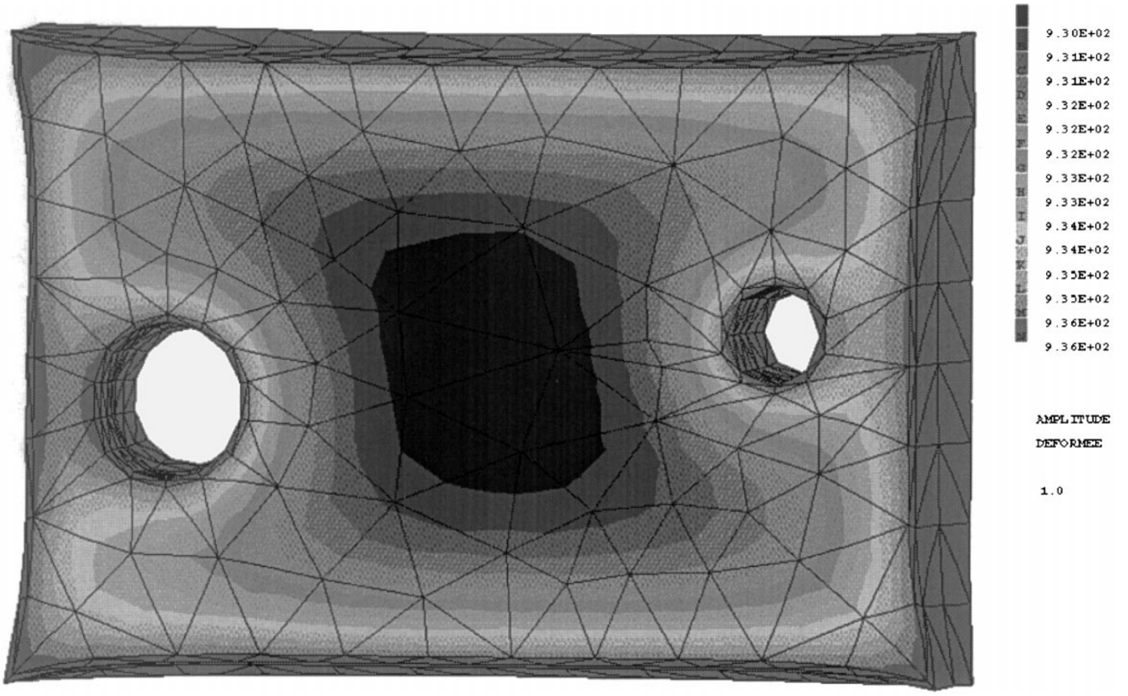

Fig. 6. Temperature gradient calculated in the validation part for 316LN powder - View from the centre of the part.

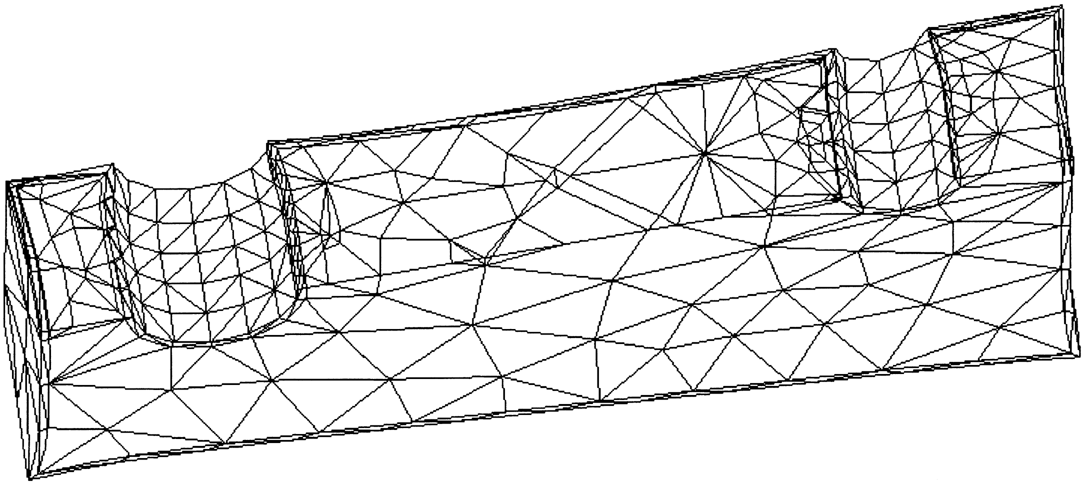

Fig. 7. Final shape of the validation part for 316LN – Simulation with PRECAD®.

Table 2
Comparison between modelling results and isotropic assumption for 316LN powder

Dimension	Displacement (in mm)	Error using isotropic assumption [6]	Error using 3D-modelling
A	17.780	−0.411	0.010
B	25.758	−0.106	0.033
I	10.363	0.446	−0.107

to be a very helpful way to optimise such parameters. It has been shown on the validation part that the accuracy of the modelling is better than the isotropic assumption. Modelling is the only way to forecast non-isotropic deformations such as enlargement of the tubes in the middle plane. This has also been shown for martensitic steel F82H in [8]. In the case of the ITER Test Module, the mock-up thickness is very close to the real thickness of the blanket, so the HIP cycle used is optimized for both cases.

5. Irradiation test on HIP materials

Powder HIPped material has potential benefits and disadvantages due to their particular microstructure and production route. It is important for the design and construction of ITER and its successors to know how these types of materials behave in response to neutron irradiation. To investigate this ECN and CEA have started to collaborate on several small scale irradiation experiments.

5.1. HIPped 316LN

Specimens of HIPped 316LN have been irradiated by ECN up to 2.5 dpa at 325°C in the HFR reactor in Petten. The tensile tests show behaviour much like 316LN-ERH plate material, irradiation hardening and reduction of ductility are similar. Only the reduction of area is significantly smaller than for plate material, but this is also observed in the unirradiated condition. Some scoping static fracture toughness tests have also been performed after 2 dpa 80°C irradiation in water. The results are shown as follows.

More details on irradiation testing of hipped 316LN are presented in a paper at this conference [9].

5.2. HIPped F82H

Limited numbers specimens fabricated of hipped F82H(mod) have been, or are being prepared to be, put into HFR scoping irradiations by ECN. Tensile and low cycle fatigue specimens have been included in a 2.5 dpa 300°C irradiation, testing of these specimens will be completed in the second half 1998. Some other specimens are planned to be included in a higher temperature (500°C) irradiation, to start by the end of this year. The results will come available in the course of 1999.

6. Conclusion

The process of powder HIP is available for manufacturing structural materials for fusion reactor such as 316LN stainless and F82H martensitic steel. The large non-isotropic deformations occurring during the consolidation of the powder are forecast with PRECAD® modelling tool. This enables the designer to optimise the initial geometry of the canister and the tools used to shape the powder. This tool is also useful to adapt the HIP cycle parameters to assure full consolidation of powder. A material data base for fusion reactor structural material (316LN stainless steel and F82H martensitic steel) is available in PRECAD/B. It has been shown previously that this powder materials have mechanical properties very close to the same composition forged material used for nuclear applications, and it is confirmed by the irradiation results developed in this paper.

References

[1] C. Dellis, G. Le Marois, J.M. Gentzbittel, G. Robert, F. Moret, J. Nucl. Mater. 233–237 (1996) 183.

[2] M. Abouaf, J.L. Chenot, J. Méca. Théor. Appl. 5 (1986) 121.

[3] C. Dellis, D. Abondance, R. Baccino, F. Bernier, F. Moret, PRECAD®, a computer assisted design and modelling tool for powder precision moulding, Proceedings of PM^{2}TEC'96, vol. 2, Part 7, 1996, pp. 289–302.

[4] O. Bouaziz, C. Dellis, P. Stutz, Creation of a material data file for modelling of an austenitic stainless steel, Proceedings of the International Workshop on Modelling of Metal Powder Forming Processes, Grenoble, 1997, pp. 67–75.

[5] D. Abondance, PhD thesis, UJF-INPG, Grenoble University, 1995.

[6] W.B. Eisen, O. Bouaziz, C. Dellis, 3D HIP modelling: Results on non-axisymmetric parts, Proceedings of the International Workshop on Modelling of Metal Powder Forming Processes, Grenoble, vol. 334, 1997, pp. 325–334.

[7] D. Bouvard, O. Bouaziz, C. Dellis, A.G.K. Jinka, Proceedings of HIP'96 Conference, ASM International, 1996, pp. 53–57.

[8] M.A. Fütterer, B. Bielak, J.P. Deffain, C. Dellis, L. Giancarli, A. Li Puma, C. Nardi, J.F. Salavy, K. Schleisiek, J. Szczepanski, Design development and manufacturing sequence of the European water-cooled Pb–17Li test blanket module, Proceedings of the ISFNT4 Conference, Tokyo, 1997.

[9] E.V. van Osch, M.I. de Vries, M.G. Horsten, these Proceedings.