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Journal of Nuclear Materials 307–311 (2002) 540–543

journal of nuclear materials

www.elsevier.com/locate/jnucmat

The effect of hot isostatic pressing parameters on microstructure and mechanical properties of Eurofer powder HIPed material

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Abstract

The production of reduced activation ferritic/martensitic (RAFM) steel by powder metallurgy and high isostatic pressing (HIP) offers numerous advantages for different nuclear applications. The objective of this work is to optimise the Eurofer powder HIP process in order to obtain RAFM solid HIPed steel with similar mechanical properties to those of a forged material. Starting from the forged solid Eurofer steel batch, the material is atomized and the Eurofer powder is characterized in terms of granulometry, chemical composition, surface oxides, etc. Different compaction HIP cycle parameters in the temperature range (950–1100 °C) are tested. The chemical composition of the HIPed material is comparable to the initial forged Eurofer. All the obtained materials are fully dense and the microstructure of the compacted material is well martensitic. The prior austenite grain size seems to be constant in this temperature range. The mechanical tests performed at room temperature reveal acceptable hardness, tensile and Charpy impact properties regarding the ITER specification.

1. Introduction

Eurofer is the European reduced activation ferritic/martensitic (RAFM) steel developed for different nuclear applications and especially for some DEMO blanket components. The powder high isostatic pressing (HIP) technology has been identified as a promising manufacturing technique for complex blanket modules [1]. Its main advantages are: (i) production of near net shape parts without melting and avoiding the severe segregation associated with melting; (ii) lower cost through a strong reduction of machining and the number of welds. In the frame of the European Fusion Technology Programme, a task of qualification of the powder HIP process, including Eurofer powder fabrication and HIP compaction parameters has been launched in order to demonstrate the potentiality of this technique. This paper presents the global process and more precisely the effect of HIP conditions on the properties of the consolidated material.

2. Materials

Eurofer steel batches were produced by Böhler following the ITER specifications. A piece of Eurofer (heat D83344) was transformed into powder by atomisation. The steel was melted in an induction furnace and an inert gas jet disintegrates a stream of molten alloy into droplets that solidified at a high cooling rate. Gas atomisation usually gives spherical particles and a log-normal size distribution. The chemical composition of Eurofer powder and the initial Eurofer heat are given in Table 1.

The chemical compositions of the powders are comparable to those of the cast material, except for the oxygen and nitrogen content. There is a low loss of

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0022-3115/02/$ - see front matter
PII: S0022-3115(02)00972-8

Table 1
Chemical analysis of heat D83344 and Eurofer powder

Element	Fe (wt%)	C (wt%)	Cr (wt%)	W (wt%)	V (wt%)	Ta (wt%)	Mn (wt%)	Si (wt%)	Ni (wt%)	O (ppm)	N (ppm)	P (ppm)	S (ppm)
Heat D83344	Balance	0.11	8.97	1.06	0.19	0.13	0.42	0.08	0.05	8	28	60	40
Powder < 45 μm	Balance	0.09	8.88	1.14	0.19	0.09	0.38	0.09	0.14	550	240	56	50
Powder > 45 μm <250 μm	Balance	0.086	8.99	1.16	0.19	0.1	0.37	0.08	0.09	90	240	21	45

carbon but no loss of other major elements. The higher oxygen content of the small particles can be explained by their higher specific surface compared to medium size particles. The large particles are scrapped in order to eliminate major contaminants. For this work we kept only medium particles in order to limit the oxygen content and to control the powder filling. These particles present a spherical shape and their metallographic observations show a homogeneous structure (Fig. 1). No local chemical analysis of the powder was performed.

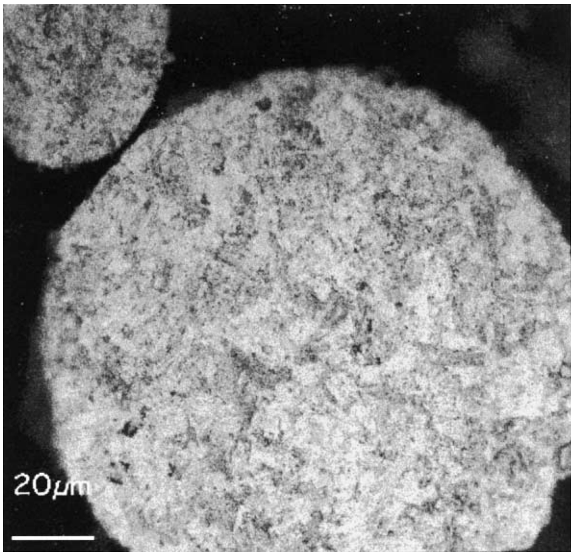

Fig. 1. Metallographic observation of a particle after atomisation.

The powder is filled into a steel canister under vibration to improve the mixing of the powder and the tap density. A minimum tap density value of 62% is required. The canister is degassed under secondary vacuum at medium temperature and sealed by welding. The canisters used in this study have an 80 mm diameter and a height of 140 mm.

3. Powder HIP consolidation

HIP is obtained by the simultaneous application of heat and pressure. Different HIP temperatures and dwell times were investigated, the pressure was fixed at 140 MPa for all tests, the HIP parameters are given in Table 2.

Table 2
HIP parameters tested

Temperature (°C)	950	950	1000	1040	1100
Time (h)	1	10	5	4	2
Pressure (MPa)	140	140	140	140	140

The imposed heating rate was 8 K/min and the cooling rate was limited by the facility and the canister size to 15 K/min. The temperature was controlled at the surface of the canister. Further HIP at 1040 °C with higher cooling rates was performed in order to evaluate the gain and the acceptable cooling rate range in which the martensitic microstructure is obtained.

4. Microstructure of HIPed material

After HIP consolidation all the materials are fully dense and present a martensitic structure as shown by hardness measurements. The hardness results of raw HIPed material (Table 3) indicate that even if the cooling rate of HIP is low, the martensitic structure is obtained in all cases. Nevertheless, the higher the HIP

Table 3
Hardness results on Eurofer HIPed material (HV30)

Heat treament	HIP conditions				
	950 °C/1 h	950 °C/10 h	1000 °C/5 h	1040 °C/2 h	1100 °C/2 h
As HIPed	422	409	392	396	378
As HIPed + Tempering 750 °C/2 h	226	215	223	220	220

temperature, the lower the martensitic hardness. The tempering time effect on the hardness results shows that the stabilization of the microstructure is only obtained between 1 and 2 h.

The microstructure was observed in the tempered state (tempering at 750 °C/2 h). The prior particle boundaries (powder outline) are clearly identified on the HIP consolidation performed at 950 °C. Large particles (oxide or precipitates) appear like a continuous line after 1 h HIP and like a dotted line after 10 h HIP dwell time at 950 °C. At higher HIP temperature, these defects at the PPB (prior particle boundaries) are less observed.

The prior austenite grain size is not always easy to define, but it seems that the prior one's size is about 8–10 μm except for 1 h HIP at 950 °C and 2 h at 1100 °C. At the highest HIP temperature the grain size is close to 10 μm. The dispersion of grain sizes and the precision of the optical observations do not allow to quantify a substantial evolution of the austenite grain size in this consolidation temperature range (950 °C/10 h to 1040/4 h). Some TEM observations were performed to check the optical grain size determination. The examination of all the HIPed materials revealed the same microstructure. The microstructure of tempered materials (2 h at 750 °C) after HIP shows martensite laths with carbides and precipitates (Fig. 2). No ferrite was detected.

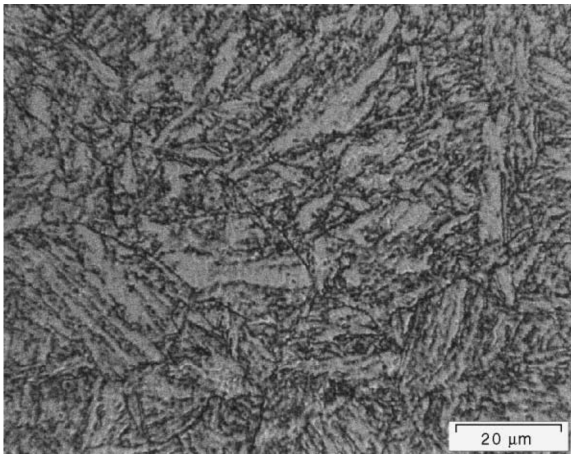

Fig. 2. Microstructure of Eurofer powder HIPped at 1040 °C and tempered at 750 °C/2 h.

5. Mechanical properties

Impact and tensile tests were performed at room temperature. The tensile and Charpy specimens were machined parallel to the canister axis. The HIPed materials were tested in the following metallurgical conditions:

Table 4
Impact properties KCV in J/cm^2 at room temperature

Heat treament	HIP conditions				
	950 °C/1 h	950 °C/10 h	1000 °C/5 h	1040 °C/2 h	1100 °C/2 h
HIPed + Tempering*	134	187	210	228	267
HIPed + PNT** + Tempering*		192	248	276	321

*750 °C/2 h.
**Post normalisation treatment 950 °C/1 h water quenched.

Table 5
Tensile results at room temperature, strain rate 4×10^{-4} s^{-1}

HIP conditions	Tempering treatment	Yield stress 0.2% (MPa)	UTS (MPa)	Uniform elongation (%)	Total elongation (%)	Reduction of area (%)
950 °C/1 h	2 h at 750 °C	530	667	3.5	13	55
950 °C/10 h	2 h at 750 °C	503	632	7.7	17	72.5
	PNT** + T*	512	636	6.5	15.5	73
1000 °C 5 h	2 h at 750 °C	541	654	6.1	14.4	67
	PNT** + T*	510	630	7.0	15.8	70
1040 °C/2 h	2 h at 750 °C	490	620	7.5	19.4	76.5
	PNT** + T*	504	613	7.8	19.5	75.5
1100 °C/2 h	2 h at 750 °C	502	615	7.5	18.5	77.5
	PNT** + T*	505	620	7.6	19.3	72.5

*Tempering 750 °C/2 h.
**Post normalisation treatment 950 °C/1 h water quenched.

– as HIPed and tempered at 750 °C/2 h (as the standard tempering temperature and time)
– as HIPed + normalisation 1 h at 950 °C/water quenched (called post normalisation treatment (PNT)) + tempering at 750 °C/2 h.

The PNT was applied in order to smooth out the various initial HIP cooling rates induced by the different HIP facilities and block sizes used in this work. The impact tests results are presented in Table 4.

The impact test results on powder HIPed materials reveal higher impact properties for the materials consolidated at 1100 and 1040 °C compared to those at 950 °C. The impact properties for 1100 °C HIP consolidation temperature followed by a post normalisation treatment reached high value 320 J/cm^2, equivalent to forged Eurofer (302 J/cm^2) with the same heat treatment (quenched and tempered). The post heat treatment improves the impact properties and different parameters could have an effect. Tensile tests were performed at room temperature at a strain rate of 4×10^{-4} s^{-1}. The tests results are reported on Table 5.

The HIP temperature effect on the tensile properties is lower than for impact properties and the post normalisation treatment play more a rule of softening. The standard powder HIPed materials present acceptable strength and ductility properties.

6. Conclusion

Eurofer cast alloys were atomised and different HIP consolidation conditions were tested. The chemical analysis does not reveal a large deviation in chemical composition between cast alloys and atomised powder. The consolidation process parameters examined were: the powder size, the filling process and the different HIP cycles temperatures ranging (950–1100 °C). All the HIPed materials are fully dense and well martensitic. The microstructure observations do not reveal an substantial prior austenite grain size evolution following the HIP temperatures. The mechanical results reveal acceptable impact and tensile properties but further work is in progress in order to improve the ductility.

Reference

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