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# Effects of the forming processes and $Y_2O_3$ content on ODS-Eurofer mechanical properties

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A B S T R A C T

This work investigates the effects of manufacturing processes on the behaviour of ODS alloys based on the Eurofer composition (Fe–9%CrWVTa). Materials were produced by mechanical alloying with two fractions of $Y_2O_3$ strengthening particles (0.3% and 0.5% in weight). The ODS powders were consolidated by hot isostatic pressing (HIP) and hot extrusion (HE). In case of HE, two distinct extrusion dies geometry were used to obtain circular or rectangular cross-sections bars. Cold rolling (CR) trials were also applied to rectangular cross-section bars to obtain 1 mm thick plates with a high cumulated cold work level ($\varepsilon$ = 80%). Microstructure, impact and tensile properties of selected samples are compared to assess the effects of the consolidation process (HIP or HE), the $Y_2O_3$ content and the shape of the extrusion die.



## 1. Introduction

This work is focused on experimental Fe–9Cr ODS steels which offer the potential of adequate high temperature strength and ductility, isotropic microstructure and mechanical properties. Their development was promoted for long-life cladding tubes of the advanced fast reactor fuel elements [1,2] and also for application in blanket concepts at operating temperature in the range 550 °C to about 650 °C [3].

The consolidation of mechanically alloyed ODS powders prepared at laboratory scale was mainly performed by HIP [4–7] while for industrial production such as MA956, MA957 or PM2000 alloys the HE was often favoured [1]. Compared to HIP, this process presents the advantage to induce a more homogeneous and finer microstructure without porosity which is of interest to optimise the material properties and in particular to decrease the Ductile–Brittle Transition Temperature (DBTT) values. Usually, the extrusion was carried out in a round-section-die [1] and the yttria content is specified in the range 0.2–0.5% in weight [1,5–7]; the both effect of the shape of the die and the increase of the yttria content (up to 0.5% in weight) are investigated here.

## 2. Materials and manufacturing procedures

The ODS Fe–9Cr based on Eurofer steel used was developed and fabricated in the context of the European Fusion Technology program (EFDA). This alloy was obtained by using mechanically alloyed powders prepared by mixing Eurofer atomized powders and $Y_2O_3$ particles under argon atmosphere at Forschungszentrum Karlsruhe (FZK).

The chemical compositions of powder batches – respectively with 0.3% $Y_2O_3$ (LXN 0449) and 0.5% $Y_2O_3$ (LXN 0450) – are presented in Table 1 and compared to the Eurofer 97 product.

A part of these powders was consolidated by HIP at Plansee (Austria).

An other part was consolidated by HE was at CEA Saclay at a temperature of 1100 °C by using a hydraulic press with a capacity of 13000 bars. For this operation, the ODS powders were encapsulated in a soft steel can and pre-heated for 1 h at the extrusion temperature. Depending of the extrusion die used, round shaped bars of 13 mm in diameter and rectangular bars of 5 mm thickness and 24 mm width were obtained which corresponds to approximately the same section reduction ratio of ~19. Rectangular bars have been cold rolled up to 3.5 mm (30% reduction in thickness) to allow the machining of samples for mechanical tests. An homogenisation heat treatment at 1050 °C for 15 min followed by a furnace cooling has been applied to all the extruded bars. Thin plates of 1 mm in thickness were successfully obtained by severe cold rolling (cumulated Cold Work (CW) of about 80%) and no cracks were detected. An homogenisation heat treatment at 1050 °C for 15 min followed by a water quench and an annealing for 1 h at 750 °C under vacuum was applied on these cold rolled specimens before tensile tests.

## 3. Experimental results

### 3.1. Microstructure and hardness of hot extruded ODS steels

Fig. 1 shows the optical micrographs obtained in the transverse cross-section of the extruded 0.3% $Y_2O_3$ ODS steels. After HE and

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**Table 1**
Chemical composition of the ODS powders (in wt%).

| Material | C | Si | Mn | Ni | Cr | V | W | Mo | Ta | O | Y | N |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Eurofer 97 ref. | 0.115 | 0.06 | 0.41 | 0.04 | 8.69 | 0.175 | 1.26 | <0.002 | 0.12 | 0.001 | | 0.019 |
| LXN0449 –0.3% $Y_2O_3$ | 0.076 | 0.13 | 0.33 | 0.07 | 8.94 | 0.18 | 1.18 | <0.005 | 0.11 | 0.127 | **0.17** | 0.022 |
| LXN0550 –0.5% $Y_2O_3$ | 0.077 | 0.12 | 0.33 | 0.06 | 9.52 | 0.18 | 1.15 | <0.005 | 0.11 | 0.176 | **0.27** | 0.026 |

annealing an homogeneous microstructure is revealed constituted by fine grains of a few microns size (Fig. 1(a) and (b)). The hardness values are measured in the range 249–265 HV5 with a larger dispersion for products extruded in a rectangular die. A refinement of the microstructure is observed for severely cold rolled sample (Fig. 1(c)) with an increase of the hardness values up to 369 HV5 which is slightly inferior to the maximal value obtained for as-extruded condition in a rectangular die (before heat treatment).

Other examinations in longitudinal direction for extruded samples with 0.5% $Y_2O_3$ reveal that an increase of the $Y_2O_3$ content has no hardening effect on the matrix but results in an increase of carbides or oxides alignments in the extrusion direction.

### *3.2. Impact properties of materials consolidated by HE or HIP*

To compare the impact properties of the ODS-Eurofer 0.3% $Y_2O_3$ consolidated by both methods (HE and HIP), the same homogenisation treatment at 1050 °C during 15 min followed by a furnace cooling has been applied to a rod bar with 67 mm in diameter consolidated by HIP at Plansee. Impact tests were performed on Charpy V-notch subsize specimens (KLST: 27 mm long, 4 mm wide and 3 mm thick), machined in longitudinal direction. Tests were conducted over the temperature range −100 to +325 °C to produce the full energy transition curves.

The transition curves showing the effect of process (consolidation route and die-geometry) and yttria content on impact properties are presented in Fig. 2.

Extruded ODS-Eurofer 0.3% $Y_2O_3$ has a lower DBTT value (−40 °C) and a higher USE (9J) than the hipped material (+70 °C and 6J). This result confirms the interest to use HE to improve the DBTT of the material. The yttria content does not affect the DBTT which remains about −40 °C but the USE decreases slightly to reach the value of 6.5J for yttria content of 0.5%. The same modification of the behaviour is observed on rectangular bars compared to round shaped: the DBTT are approximately the same (−40 °C) but a decrease of the USE (6J) is measured. In addition, an other result (not reported in the Fig. 2) gives a lower value for the USE (3J) in the case of a specimen with 0.5%$Y_2O_3$ content extruded in a rectangular die. It should indicate that an increase of the $Y_2O_3$ content and the use of a rectangular die lead to a decrease of the USE. This result also indicates that the extrusion ratio is not the single parameter which determines the local deformation. The fields of flow rate are not the same in round or rectangular extrusion and result in different consolidation threshold.

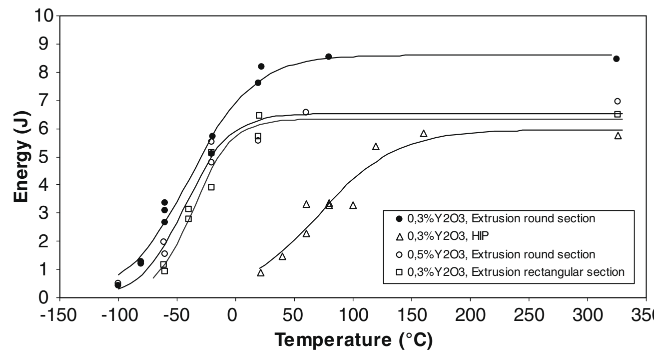


**Fig. 2.** Effect of forming process and yttria content on impact properties.

### *3.3. Tensile properties of materials consolidated by HE*

Tensile properties were performed on cylindrical specimens of 2 mm diameter and 12 mm gauge length except for severely cold rolled materials for which plate specimens of 1 mm of thickness, 2 mm of width and 26 mm of total length were used. Specimens have been machined in longitudinal direction of the bars. Tests were conducted in the temperature range 20–750 °C with a $7.10^{-4}\,s^{-1}$ strain rate. For each test temperature, the 0.2% Proof Stress, the UTS (Ultimate Tensile Strength) and the total elongation were determined. Two specimens were tested for each condition and a good reproducibility was found.

The comparative evolution of the tensile properties as a function of the test temperature is reported on Fig. 3. The yield stress and the UTS of the alloys with 0.3% or 0.5% $Y_2O_3$ contents extruded in round and rectangular section are roughly the same. The total elongation is around 20% for all the samples after HE and heat treatment but a peak of ductility is observed at 550 °C for ODS-Eurofer 0.5% $Y_2O_3$ (At ∼30%) and at 650 °C for ODS-Eurofer 0.3% $Y_2O_3$ (At ∼25%).

For the severely cold rolled sample a very important increase of the tensile strength is observed in the temperature range 20–500 °C (the measured values of UTS and $Rp_{0,2}$ are 300 MPa above those measured for as-extruded conditions) but the total elongation is significantly lower with a difference of almost 10% at 450 °C. Beyond the gap between as-extruded and severely cold rolled samples decreases to reach equivalent level of strength and ductility at 600 °C and above.

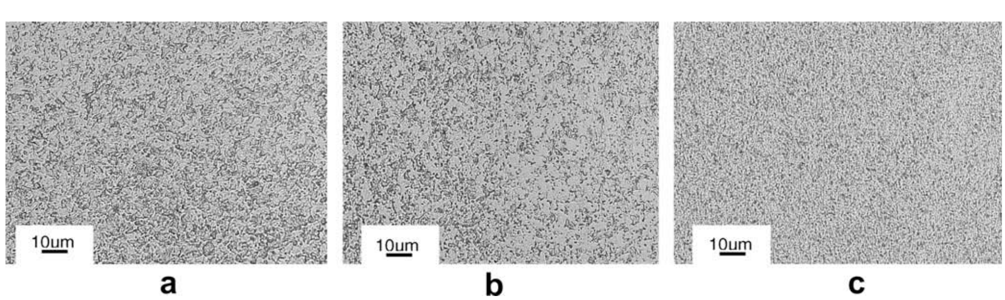


**Fig. 1.** Optical micrographs showing the microstructure of the 0.3% $Y_2O_3$ ODS steel (a) HE round section (b) HE rectangular section and (c) HE rectangular section + CR ($\varepsilon$ = 80%).

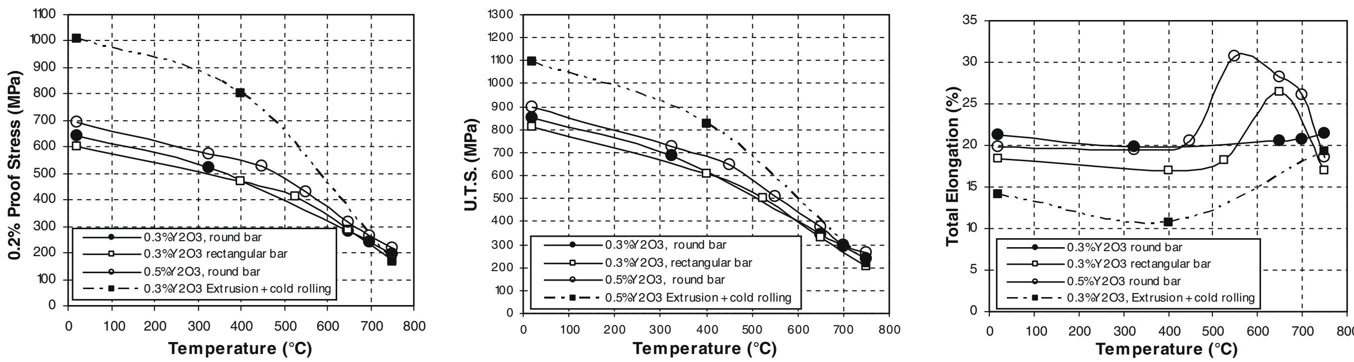


**Fig. 3.** Effect of die-geometry and yttria content on the tensile properties.

As the cold rolling tests were carried out in the extrusion direction, a very elongated fine grain morphology in this direction is expected. This grain structure can leads to an anisotropic behaviour and it should be useful to check the mechanical properties in the transverse direction.

## 4. Conclusions

The mechanical properties of ODS-Eurofer alloys containing 0.3% and 0.5% $Y_2O_3$ obtained by using different forming processes have been characterised.

- The impact tests confirm the best behaviour of extruded specimens in longitudinal direction, which display lower DBTT (−70 °C) and higher USE (9J) compared to HIP samples (DBTT = +70 °C, USE = 6J). As extruded materials are presumably less isotropic compared to HIP materials, further tests should be interesting to compare their behaviour in transverse direction.
- For the same processing conditions, an increase of the yttria content from 0.3% to 0.5% leads to a slight improvement of the tensile properties, DBTT is not affected but a decrease of the upper shelf-energy is observed.
- The use of a rectangular die in place of a round shape one has no influence on the tensile properties but results in a decreasing of the upper shelf-energy values.
- Specimens machined from plates with high degree of CW exhibit the highest level of UTS and $Rp_{0,2}$ but lower ductility compared to as-extruded materials.

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## References

[1] S. Ukai, T. Narita, A. Alamo, P. Parmentier, J. Nucl. Mater. 329–333 (2004) 356–361.
[2] A. Alamo, V. Lambard, X. Averty, M.H. Mathon, J. Nucl. Mater. 329–333 (2004) 333–337.
[3] R. Lindau et al., Fusion Eng. Design 75–79 (2005) 989–996.
[4] R. Lindau et al., J. Nucl. Mater. 307–311 (2002) 769–772.
[5] V. de Castro et al., J. Nucl. Mater. 322 (2003) 228–234.
[6] C. Cayron et al., J. Nucl. Mater. 335 (2004) 83–102.
[7] Z. Oksiuta, P. Olier, Y. Decarlan, N. Baluc, in: Proc. of EURO PM2007, vol. 2, Toulouse, 15–17 oct., 2007, pp. 101–108.