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Mechanical properties of several newly produced RAFM steels with Tungsten content in the range of 2 wt%

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ABSTRACT

The contribution of ENEA together with Rina-CSM to the Eurofusion programme “WPMAT-Advanced Seels” deals with the development of innovative RAFM steels able to withstand the critical temperatures typical of the different operational environments foreseen for the blanket of the first DEMO reactor. The optimization of the chemical composition and the Thermo Mechanical Treatment for these materials should be done according to the blanket operating temperatures that are related to two possible working conditions: the WCLL-BB (Water Cooled Lead Lithium Breeding Blanket) or the H(D)CLL-BB (Helium (Dual) Cooled Lead Lithium Breeding Blanket). On the one hand the “water-cooling” option implies a minimum irradiation temperature for the blanket material in the range of 280–350 °C. On the other hand, the “helium-cooled” and the “dual-coolant” solutions imply an operating temperature for the blanket material in the range of 650 °C. Therefore in the first case the target is the improvement of the toughness of the martensitic alloys; whilst concerning the second scenario the target is the development of more creep resistant martensitic steels, suitable to tolerate such a high operating temperature. In both the cases the Tungsten content plays a key role, both in terms of solid solution hardening and influence on the DBTT. Two alloys aimed at fulfilling the specifications for the two DEMO operating conditions, both with increased Tungsten content respect to Eurofer, have been produced and characterized. The mechanical properties of these two alloys are hereby reported and discussed.

1. Introduction

Reduced Activation Ferritic Martensitic (RAFM) are being widely developed in Europe (EUROFER), Japan (F82H and JLF-1), Russia, China (CLAM), the USA (9Cr-2WVTa by ORNL) and India. Comprehensive reviews and comparisons among these steels concerning mechanical properties are reported in [1–8]. The contribution of ENEA together with Rina-CSM to the Eurofusion programme “WPMAT-Advanced Seels” deals with the development of innovative RAFM steels, with limited variation in chemical composition respect to the reference one of EUROFER (9% Cr, 1% W), able to withstand the critical temperatures typical of the different operational environments foreseen for the blanket of the first DEMO reactor. On the one hand the “water-cooling” scenario implies a minimum irradiation temperature for the blanket material in the range of 280–350 °C. Therefore, due to the irradiation behaviour of EUROFER, namely to the DBTT shift under irradiation, the target is the development of much tougher alloys, suitable to tolerate the low irradiation temperature. On the other hand, the “helium-cooled” and the “dual-coolant” options imply an operating temperature for the blanket material in the range of 650 °C. Therefore the high temperature behaviour of the proposed innovative alloys should be improved, namely the target is the development of more creep resistant martensitic steels, suitable to tolerate such a high operating temperature. In both the cases the Tungsten content is thought to be a key issue as this element acts as “solid solution hardener” and then it is expected to improve the mechanical properties, even at high temperature, and consequently the creep resistance as well. Moreover, standing to recent results, just achieved in the frame of the Eurofusion project [9], an increase of this element content to a range of 2 wt% also seems to provide some beneficial effects in terms of toughness of the produced RAFM steels; namely higher variability in the DBTT and susceptibility to the tempering temperature was noticed in the casts with doubled Tungsten content respect to Eurofer. Therefore two variations in chemical composition respect to the reference Eurofer 97/2 have been produced and characterized, both with increased Tungsten content (1,8/1,9 wt%); the first (the 3125 alloy) is conceived for the

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low temperature applications (WCLL-BB); the second one (the 3126 alloy) is proposed for the high temperature applications (HC/DCLL-BB). The mechanical properties of these two alloys are hereby reported and discussed.

2. Experimental

The main guidelines adopted at the alloy design stage have been the following ones:

- Increase the solution hardener element (W) in order to enhance high temperature resistance and, more specifically, creep strength in the high temperature applications;
- Avoid exceeding a Carbon content of 0,13 wt% in order to hinder the precipitation of $M_{23}C_6$ and to foster the precipitation of V and Cr nitrides whose dimensions are expected to be lower than the previous ones;
- Achieve a 25% increase of primary precipitation in γ field during the austenitizing (according to the total amount of TaC precipitation assessed by Thermocalc up to 1100 °C) aimed at limiting the prior austenite grain size enlargement in order to conserve a sufficient toughness;
- Avoid δ-region which could cause an undesired loss of toughness;
- Achieve a large 100% γ field because of workability reasons; namely the γ field should be large enough in order to perform very high temperature thermal treatments to chemically homogenize the alloy.

With these targets, we investigated several ranges of composition from a thermodynamics point of view using THERMOCALC with TCFe7 database and JMATPro codes, and precisely: $8.5 \leq Cr \leq 9$ wt%, $1.5 \leq W \leq 2$ wt%, $0 \leq V \leq 0.3$ wt%, $0.05 \leq C \leq 0.15$ wt%, $0.05 \leq Ta \leq 0.2$ wt%, $0 \leq N \leq 0.05$ wt%. The chemical compositions which have been eventually casted VIM (Vacuum Induction Melting) are reported in the next table (Table 1). The 3125 and the 3126 ingots have been casted VIM by RINA Consulting – CSM, in lab scale (80 kgs each ingot). Ni, Mo, Cu, Nb, Al, B, Co, P and S have been kept as low as possible.

The applied Thermo-Mechanical Treatments (TMT) are the double austenitization and the "ausforming" for the low and high temperature applications respectively, as reported in the next table (Table 2). Previous works [10–12] have already demonstrated the effectiveness of the multiple austenitization stages in terms of Prior Austenite Grain Size (PAGS) refinement and homogeneization. The constrain on the DBTT has driven the choice of the austenitization temperature so as to maintain the PAGS in an acceptable range. The approach in the choice of the austenitization temperature has been different for the two alloys;concerning the 3125 alloy, the austenitization temperature has been determined after optimization (Scanning Electron Microscopy observations) on the PAGS in order to keep this parameter in the range of 10 µm. Concerning the 3126 alloy instead, the austenitization temperature should be high enough to put into solution as much carbide or nitride forming elements as possible, but avoiding an excessive PAG growth. A medium PAGS, namely in the range of 40–50 µm, is thought to be sufficient for creep and, at the same time, not so detrimental for toughness. The ausforming treatment is known to improve creep resistance by "dislocation pinning" through precipitation [13–22]. In this case it has been applied with two different hot working (h.w.) ratios on the 3126 alloy. The tempering temperature has been fixed, according to common practice, as best compromise between toughness and tensile properties. The reason for the lower tempering temperature adopted on the 3126 alloy is related to the more demanding specifications on tensile properties concerning the high temperature applications.

Table 1
Chemical compositions (contents in wt%).

Heat	Cr	C	Mn	V	W	Ta	N
Eurofer 97/2	9	0,11	0,5	0,2	1	0,12	0,02
3125	9	0,12	0,1	< 0,05	1,8	0,15	0,002
3126	8,7	0,13	0,49	0,25	1,9	0,14	0,032

Table 2
Thermo-mechanical treatments applied on the two alloys.

Heat	Austenitization (°C-hrs)	Austenitization (°C-hrs)	Ausforming (°C – h.w.%)	Tempering (°C – hrs)
3125	980	980	–	760 – 1
3126 (a)	1180	–	650 °C – 40%	700 – 1
3126 (b)	1180	–	650 °C – 60%	700 – 1

A "screening" on the mechanical properties has then been carried out on each of the proposed combination of chemical compositions with Thermo Mechanical Treatments. Tensile tests have been carried out on an electro-mechanical machine equipped with a 50 kN load cell; the geometry of the samples used in the tensile tests was cylindrical (8,9 mm gauged diameter and 54 mm gauged length). Tensile tests have been carried out controlling the crosshead displacement. An extensometer was applied on the gauged length of the sample during the tests in order to record the strain all over the test duration both at room temperature and at high temperature. The strain rate has been kept equal to $2{,}5 \times 10^{-4}$ mm/mm/s to determine the Yield Strength and further, up to the achievement and exceeding of the UTS (Ultimate Tensile Strength), according to ASTM E8/E8M:2013. For the creep tests (testing procedure in agreement with ASTM E139:11) several frames (1:10 and 1:15 lever ratio) equipped with auto-levelling arm device and three zones P.I.D. controlled furnaces have been employed. The cylindrical geometry of the samples used in the creep tests is the following: 5,6 mm gauged diameter and 30,3 mm gauged length. Impact tests have been carried out by means of both Charpy ISO-V specimens (10 × 10 × 55 mm geometry; test procedure according to UNI EN ISO 148/1:2012 on a 300 J size pendulum "Wolpert") and KLST specimens (10 × 10 × 55 mm geometry; test procedure according to DIN 50115 on a 60 J size pendulum "Ceast"). The outcomes of all these tests are reported.

3. Results

The results of the impact tests carried out on both Charpy ISO-V and KLST specimens are compared in the next plots (Fig. 1) and summarized in the next table (Table 3).

A marked difference between the outcomes of the 3126 alloy and the ones of Eurofer 97/2 and 3125 alloy is noticeable in terms of both DBTT and USE; the steel meant to be employed in the "low temperature" operating conditions (3125) is much more performing than the 3126 alloy in terms of toughness properties. This is expected and due to the different specifications for the two operating conditions; the "lowest possible DBTT" for the "low temperature" scenario; a "sufficient toughness" for the "high temperature" scenario, just in order to carry out safe manufacturing and assembling processes at room temperature for the alloys meant to be used in this last one (3126 alloy). Concerning the alloy meant to be employed in the low temperature scenario (batch 3125) we can notice a marked improvement in the DBTT, compared to Eurofer with the same double austenitization thermal treatment. The double austenitization proves beneficial by itself in shifting the DBTT as we can observe on Eurofer 97/2 [11], but this shift (respect to the standard treatment on Eurofer 97/2) appears even more when this thermal treatment is applied on the alloy with Tungsten content close to 2 wt%.

The results of the tensile tests are reported in the following plots (Fig. 2). The fitting functions are 3rd degree polynomials. The two

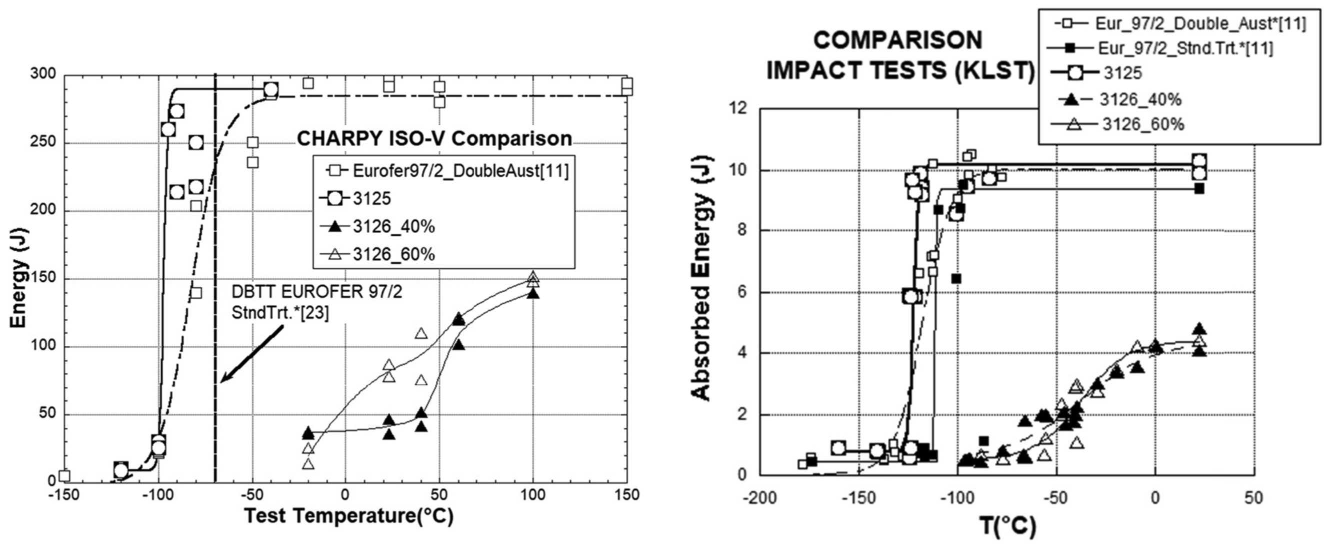

Fig. 1. Comparison between the results of the Impact Tests; Charpy ISO-V (left) and KLST (right).

graphs (Fig. 2) are meant to provide a comparison between the tensile properties (yield strength and ultimate tensile strength) of the two newly produced alloys and the ones of Eurofer 97/2 [9], recorded all over the temperature range taken into account.

Concerning the low temperature applications the tensile properties of 3125 alloy, in which the Tungsten content has been increased to 1,8 wt%, are markedly higher compared to Eurofer 97/2 (Tungsten content = 1%).

Concerning the high temperature applications, as it results from the plot, the tensile properties of 3126 alloy are again considerably improved respect to Eurofer 97/2 with the same applied ausforming treatment (40% hot working ratio at 650 °C). The different hot-working ratios applied on 3126 alloy don't take substantial differences in terms of high temperature (650 °C) resistance. We can deduce that meaningful dislocation recovery presumably occurs and leads to the annihilation of the effect of the higher dislocation density of the 60% hot worked alloy. This is also confirmed by the following creep tests.

The results of the creep tests carried out on 3126 alloy at 650 °C are compared in the next graph (Fig. 3). The achieved times to rupture are comparable to the ones of ausformed Eurofer 97/2, obtained in a previous campaign [9]. The tests carried out on 3126 alloy also confirm what was stated before about the results of the tensile tests; the hot working ratio increased to 60% doesn't provide any profit in terms of high temperature resistance.

4. Conclusions

Generally speaking, the improvement of tensile properties of RAFM alloys (at room temperature as well as at higher temperatures) is associated to the hardening of the steel due to dislocation network and precipitation effects. This effect is well noticeable on the 3126 alloy, where the fraction of secondary precipitation (which we expect to be increased compared to 3125 alloy, according to Thermocalc), accompanied by heavy hot working ratios during the ausforming treatment, results in enhanced tensile properties at the expense of poorer toughness and higher DBTT. On the one hand the 3126 alloy provides excellent creep resistance but lacking impact properties (DBTT superior to 0 °C by means of Iso-V Charpy specimens in both hot-working conditions); on the other hand, the 3125 alloy provides excellent impact properties (DBTT close to −100 °C), but worsens significantly in terms of tensile properties. In both the cases the Tungsten content in the range of 2 wt% appears beneficial as long as it increases the tensile strength respect to Eurofer 97/2 but leaves toughness unaffected or even slightly improved.

Concerning the low temperature applications, to which the 3125 alloy is addressed, the Tungsten content in the range of 2 wt%, accompanied by an increase in Ta content, proves beneficial in improving the toughness behaviour (in terms of lower DBTT) as resulting from impact tests on both Charpy Iso-V and KLST specimens. On the other hand, concerning the high temperature applications, to which the 3126 alloy is addressed, the "solution hardening" effect of Tungsten can be well noticed in the plot reporting the tensile properties. The supposed increase of TaC fraction combined with the adoption of the Ausforming TMT and the increase of the austenitization temperature are also thought to contribute to the enhancement of the high temperature resistance. The creep properties result comparable to the ones achieved with the best Ausforming TMT condition on Eurofer. The toughness is poor, as expected, but however it is thought sufficient to grant safe manufacturing and assembling processes, as long as the DBTT remains below 0 °C, at least by testing KLST specimens.

After this initial screening on mechanical properties the research work must be considered not concluded and further experimental activities are still on-going with a twofold purpose; on the one hand to further optimize toughness and microstructure of the 3125 alloy by

Table 3
Results of the Impact Tests; concerning Eurofer 97/2 from literature sources [11,23].

Heat	DBTT ISO-V(°C)	DBTT KLST(°C)	USE ISO-V(J)	USE KLST(J)	LSE ISO-V(J)	LSE KLST(J)
Eurofer 97/2 – Stnd. Trt.	−70	−111	250	9.34	10	0.46
3125 alloy – Double Aust	−97	−122	290	10.2	10	0.8
3126 alloy – Ausformed40% h.w.	52	−37.3	140	4.4	37	0.5
3126 alloy – Ausformed60% h.w.	33	−37.3	150	4.4	20	0.5

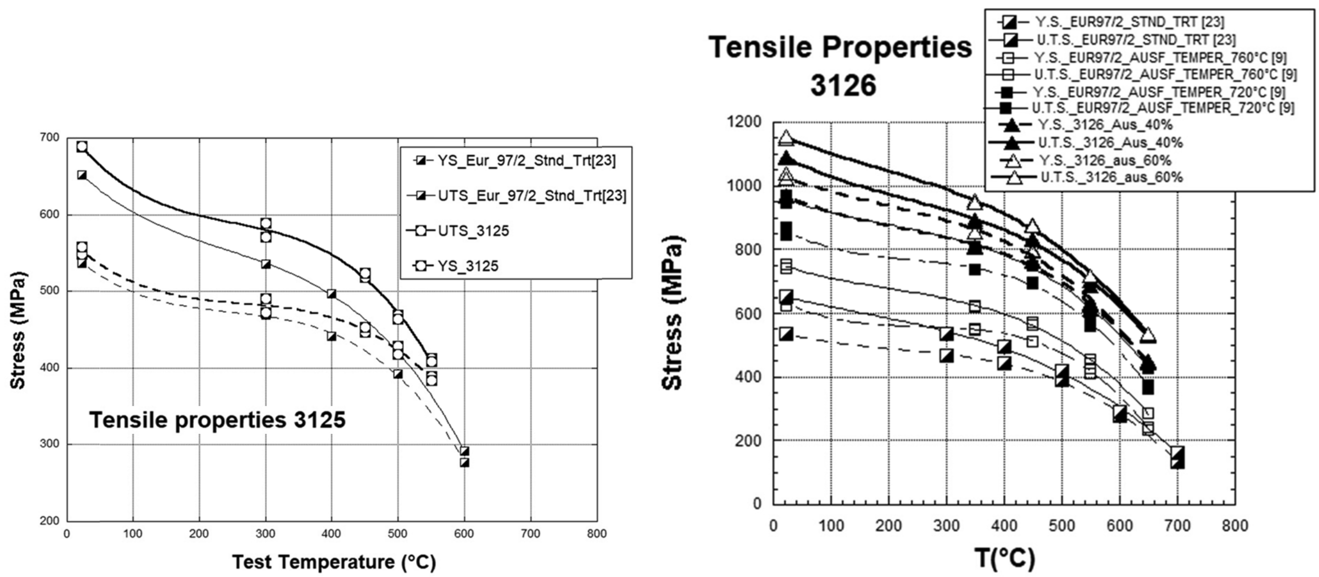

Fig. 2. Comparison among the tensile properties obtained for each alloy (Eurofer 97/2 from [9,23]); 3125 for the low temperature application (left); 3126 with the two different hot working ratios for the high temperature application (right).

applying different TMTs which have proven successful on Eurofer 97/2; on the other hand to understand which is the contribution played by each of the proposed modifications (chemical composition, austenitization temperature, TMT) on the increase of the high temperature resistance of the 3126 alloy.

CRediT authorship contribution statement

C. Cristalli: Project administration, Conceptualization, Investigation, Data curation, Visualization, Writing - review & editing. **L. Pilloni:** Supervision, Conceptualization, Investigation, Data curation. **O. Tassa:** Conceptualization, Investigation, Data curation, Resources. **L. Bozzetto:** Conceptualization, Investigation, Data curation, Resources.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

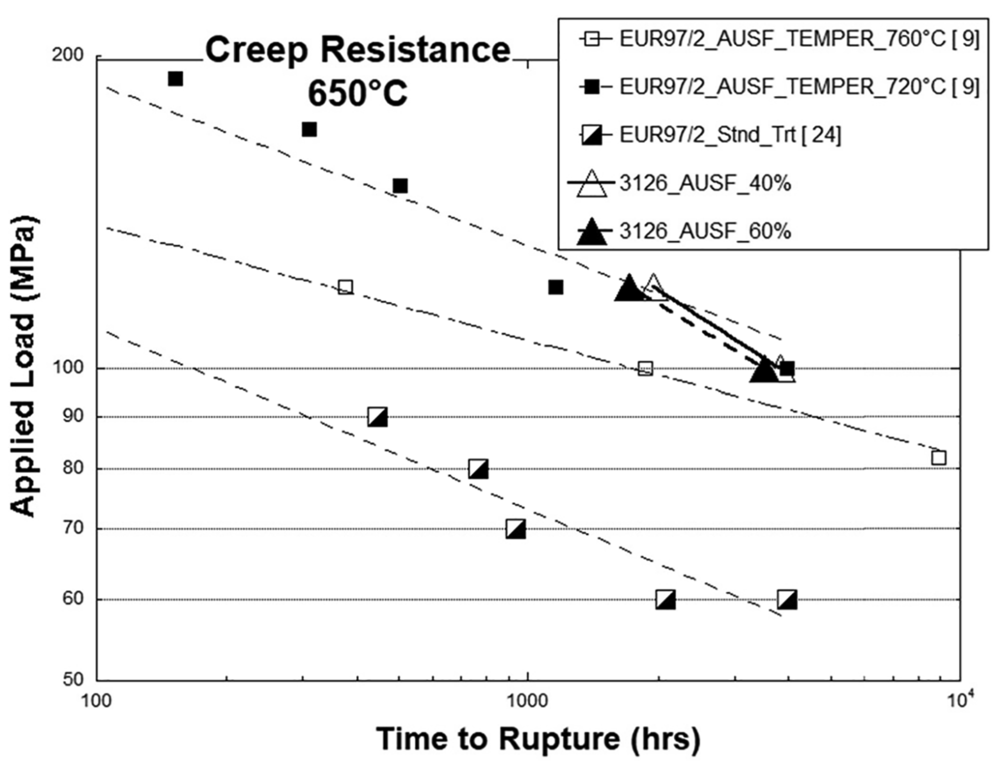

Fig. 3. Comparison of the times to rupture at 650 °C obtained for each alloy (Eurofer 97/2 from [9,24]).

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