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Innovative RAFM steels with improved impact properties

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A R T I C L E I N F O

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A B S T R A C T

The contribution of ENEA together with Rina-CSM to the Eurofusion programme "WPMAT" (Work Package MATerials) deals with the development of innovative RAFM steels able to withstand the critical low temperature (280–300 °C in the "water-cooling" option) foreseen for the blanket of the first DEMO reactor. Therefore the target is the development of tougher alloys, suitable to prevent the low temperature irradiation embrittlement. In addition to the DBTT shift due to the dpa damage under irradiation, also the issue of the increased embrittlement due to Helium production must be taken into account.

The alloy design focused on chemical composition optimization in terms of contents of some main key elements as Carbon, Manganese, Vanadium, Nitrogen and Tantalum. An overall indication deriving from the last neutron irradiation experiment was to increase Vanadium and Nitrogen at the expense of Carbon and Manganese. The ingot has then been cast with accurate control of impurities. The final product has proven compliant to the specifications concerning RAFM steels for fusion environment.

Alternative thermo-mechanical treatments have then been applied on this RAFM steel. On the one hand the DBTT after a standard double normalization and tempering treatment is definitely successful; -140 °C on KLST specimens. The extremely low DBTT must be considered a consequence of the choices done on the chemical composition. On the other hand a particular TMT, whose aim is to induce recrystallization of the laths substructure and obtain an equiaxed nano-grain structure, has been investigated. Supposed to increase the resistance to crack propagation, the decrease in grain size (less than one μm) leads to a transition curve that doesn't show a sharp drop in the absorbed energy, but instead a more gradual transition from the USE to the LSE. The tensile properties in the recrystallized condition result markedly higher than after the conventional treatment.

1. Introduction

Nowadays three main design prototypes for the construction of the DEMO reactor are considered; the HCLL (Helium Cooled Lead Lithium), the DCLL (Dual Cooled Lead Lithium) and the WCLL (Water Cooled Lead Lithium). The Italian contribution to Eurofusion is mainly focused on the WCLL blanket. In this scenario, as it can be noticed in several references [1,2], the blanket components subjected to the lower temperatures (280–300 °C) undergo an irradiation damage up to 10 dpa/fpy (displacement per atom/full power year). In addition to the DBTT (Ductile to Brittle Transition Temperature) shift due to the dpa (Displacement per Atom) damage under irradiation [3], also the issue of the increased embrittlement due to Helium production must be taken into account. This issue appears even more detrimental and less manageable because the DBTT shift due to the Helium production does not saturate with the dose, as it results from previous works reported in literature [4–6]. Moreover, even if cooling for the low temperature option of the blanket is foreseen with high pressurized water conditions at about 290 °C, the operational temperature of the structural material is expected to range from ~300 °C to ~500 °C during normal operation due to the applied power densities and the formed thermal gradients in the component. Thereby, the problem related to irradiation induced embrittlement is related to temperatures below 350 °C, but there will be also areas of the blanket structural material which will undergo markedly higher temperatures and related detrimental effects on structural material.

Recent neutron irradiation campaigns [7] revealed that an increased Vanadium and Nitrogen content at the expense of Carbon and Manganese resulted in improved behaviour (in terms of limited hardening and embrittlement) after irradiation. In the frame of this irradiation campaign an alloy (labelled as "O"), originally developed for the high temperature applications with the name of 2991 [8], underwent

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exposure in reactor up to 2,5 dpa. The limited irradiation hardening and embrittlement which resulted for this alloy, in the same range as for the other alloys taken into account in the experiment, suggest that, despite the thermal treatment applied (involving very high normalization temperature and “ausforming”) which isn’t fully appropriate for a “low temperature” operating condition, something in the chemical composition worked well under irradiation. This alloy was infact conceived according to the previously mentioned criterion, namely an increased Vanadium and Nitrogen content at the expense of Carbon. Carbon and Manganese are infact known to aggregate preferentially on interstitial loops, thus fostering irradiation hardening. A decrease in their content is then welcome. Therefore such a guideline has been followed in the alloy design of this new steel named 3387, very similar to the previously developed 2991 except for the Manganese content which has been lowered. Also the thermal treatments have been tailored this time on a “low temperature” scenario compatible with the operating conditions of the WCLL-BB. A standard double normalization (at lower temperature respect to the 2991 alloy) and tempering treatment has then been applied on this alloy.

Moreover an alternative TMT (Thermo Mechanical Treatment) has been applied on the 3387 cast alloy. In previous works [9,10] it has already been demonstrated that it is possible to refine the grain size by applying cold working-tempering treatments on the tempered Martensite. The aim of such a TMT is to induce recrystallization of the laths substructure and obtain an equiaxed nano-grain structure. The gain in grain size reduction on Eurofer 97/2 [10] was very relevant; from 10 μm of the optimized PAG (Prior Austenite Grain) to less than one micron of the ferritic grain after recrystallization. The impact properties were also encouraging; the recrystallized steel resulted much more tolerant respect to the cleavage at very low temperature, delaying the onset of the LSE (Lower Shelf Energy). Therefore this promising TMT has been applied on the new 3387 alloy as well. The multiplication of the grain boundaries achieved by means of the recrystallization treatment would likely increase the recombination sites of point defects on nano-grains boundaries under irradiation as well as the dilution of He on grain surface, delaying the formation of He bubbles on grain boundaries and, therefore, the susceptibility to the He embrittlement. In [11] infact, it has been observed as Helium bubbles accumulate preferentially on grain boundaries, thus fostering an intergranular fracture mode, and the authors state that helium lowers the critical stress for intergranular fracture. It is therefore assumed that, given a certain Helium production rate, a microstructure characterized by an increased grain boundary surface would provide a meaningful countermeasure to this “grain boundary sensitive” Helum embrittlement.

The comparison between the mechanical properties (impact and tensile tests) deriving from the standard treatment and from the recrystallization one, as well as microstructural analysis concerning grain size and precipitate distribution, are reported and discussed in this paper.

2. Experimental

A new heat with slight chemical composition modifications with respect to EUROFER 97 has been produced. The new heat 3387 has been fabricated in order to match the impurity level of nuclear grade EUROFER steel. Particular attention was put to the control of level of Carbon, Silicon and Manganese, aiming to reduce grain boundary precipitation and to improve the steel cleanliness. The aim is also to increase the Nitrogen amount (more MX precipitation expected) compatibly with a VIM (Vacuum Induction Melting) process. Given the incompatibility of Nitrogen added alloys with the VAR (Vacuum Arc Remelting) process, an "ad hoc" VIM process under nitrogen atmosphere has been developed and optimized to obtain a specific final product with the required degree of purity, without further remelting process. The main factors that have to be controlled in order to avoid the gas bubble formation are the pressure and the casting temperature. Furthermore, raw materials with very high purity have to be used in order to control the material cleanliness. Other guidelines adopted in the design of the new alloy refer to the maintenance of a large 100 % γ (gamma phase) field because of workability reasons despite Manganese reduction. Eventually, concerning Tungsten content, despite previous experimental works [12] demonstrated that an increase in this element content didn’t worsen so much impact properties, it has been considered preferable to leave this element weight percentage in the same range (1 %) as EUROFER 97/2, in order to avoid solid solution strengthening effect and consequent expected decay in toughness.

The EUROFER 97/2 plate (heat 993,391) hasn’t been produced at RINA-CSM (Rina Consulting - Centro Sviluppo Materiali S.p.A), but has been supplied by Bohler/Saarschmiede, Austria, within an Eurofusion agreement to distribute it among all the project partners.

A VIM casting (80 kg) has been fabricated, with the selected composition. The size of the ingot, casted at the foundry of RINA-CSM was the following: Diameter ≈ 150 mm, Length ≈ 550 mm, Weight ≈ 80 Kg The measured chemical composition of 3387 alloy is reported in Table 1; the target values for all the elements result matched. The content of Nitrogen is in line with the expected value, assuring a minimum presence of micro porosity. The content of all the elements not mentioned in the table (Ni, Mo, Cu, Nb, Al, B, Co) results below the impurity level, according to specification which requires their content as low as possible.

The experimental TMTs for the investigated steel, 3387, are summarized in Table 2; for comparison the conditions used for EUROFER 97/2 are reported; for this steel the results were discussed in a previous paper [10]. Concerning the recrystallization TMT, starting from the hot rolled plate, 15.8 mm thick, a second hot rolling step is performed to reduce the thickness down to 9.8 mm. This step is necessary in order to adapt the sheetmetal thickness to the one admitted at the entry of the cold rolling machine. Eventually, this plate is cold rolled according to the scheme of the investigated recrystallization TMT (50 % c.w.). The final thickness of the cold rolled plate is about 5 mm.

The 3387 cast, as well as EUROFER 97/2 received by Bohler/Eurofusion, have been treated at RINA-CSM. Concerning the austenitization stage a double normalization has been applied as this repeated heat treatment had already proven successful on EUROFER 97/2 to improve homogeneity and size of the Grain [9]. The lower austenitization temperature of 3387 compared to EUROFER 97/2 is related to the different chemical composition and likely lower amount of primary precipitation in the former respect to the latter. The lowest tempering temperature taken into account at the end of the recrystallization treatment is 550 °C. As specified in the introduction the foreseen operating temperature of the WCLL Breeding Blanket is 280–300 °C; therefore 250 °C (550–300) has been assumed as a sufficient margin to maintain stability under irradiation. There should actually be other issues related to short time temperature excursions due to local overheating of components that can raise the temperature also above 500 °C but this issue hasn’t been addressed at this stage since the main objective is now to exploit as much as possible the recrystallization effect and this implies keeping low the final tempering temperature according to previous screenings [10].

Microstructural analysis have then been performed on some selected experimental conditions. LOM (Light Optical Microscopy) analysis has been carried out after the double normalization stage in order to identify the PAGS (Prior Austenite Grain Size). In order to identify grain boundaries the samples were tempered at 680 °C/1 h TEM analysis has been performed after the final tempering of the standard treatment and after final recrystallization treatment, both on extraction replicas and

Table 1
Chemical composition of the cast ingot (elements in wt. %).

Heat	Cr	C	Mn	V	W	Ta	N
EUROFER 97/2	9	0.11	0.55	0.2	1.06	0.1	0.02
3387 alloy	9	0.05	0.04	0.3	0.99	0.05	0.07

Table 2
TMT (Thermo mechanical treatments) applied on EUROFER 97/2 and on 3387 alloy; acronyms used: CW (Cold Working); AC (Air Cooling); WC (Water Cooling).

Treatment/ material	EUROFER 97/2 (heat 993,391)	3387
Treatment before CW	Double austenitization 1020 °Cx0.5h + AC + 1020 °Cx0.5h + AC Tempering 760 °Cx1h + AC	Double austenitization 960 °Cx1h + WC + 960 °Cx1h + WC Tempering 760 °Cx30 min + AC
1st CW	Cold rolling: 50 %	Cold rolling: 50 %
Treatment after CW	Tempering: 550 °Cx1h + AC	Tempering: 550 °Cx1h + AC

thin foils, in order to deduce precipitate distribution and recrystallized grain size respectively. The TEM instrumentation is JEM-3200FS Field Emission Microscope, operated at 300 kV and equipped for EDS (Energy Dispersive Spectroscopy, Oxford Instruments Inca Link ISIS) and EELS (Electron Energy Loss Spectroscopy, Gatan Imaging Filtered). Extraction replicas were combined with high spatial resolution microanalysis of fine second phase particles, with size in the range 10 nm to 300 nm. TEM on thin foils has been performed as well on the samples which underwent cold rolling and recrystallization treatment at 550 °C x 1 h Thin foil specimens (3 mm-discs cutting from thin slices) were prepared using twin–jet electropolishing of the disk followed by electropolishing up to perforation at disk centre.

The Vickers micro-hardness has been measured with a 5 kgf load (HV5). The thickness of the final tempered plate on which the hardness measurements have been carried out is 4.9 mm. The hardness values have been measured in the middle of the plate.

Charpy Iso-V Notch (CVN) samples extracted on l-T direction (longer edge of the specimen parallel to the rolling axis of the sheetmetal) have been used to test impact properties after standard (double normalization and tempering) treatment on a 360 J Pendulum. On the recrystallized condition it hasn't been possible to perform impact tests on Iso-V specimens (10 × 10 × 55 mm) due to the narrow thicknesses of the cold worked plates.

On the recrystallized plates, as well as on the sheetmetals which underwent standard treatment, impact tests on KLST specimens (according to DIN 50,115 standard) have been carried out. Therefore KLST specimens have been employed. These specimens differ from the standard Charpy Iso-V ones for the reduced size; 4 × 3 × 27 mm^3 instead of 10 × 10 × 55 mm^3. Also the depth and shape of the notch is different; the height of the notch is 1 mm in the KLST compared to 2 mm in the Charpy Iso-V. KLST specimens have been extracted in l-T direction and tested with a 25 Joule mini pendulum (test procedure according to DIN 50,115).

Tensile tests have been conducted according to UNI EN ISO 6892–1:2020 on cylindrical specimens extracted in T-L orientation (axis of the specimen perpendicular to the rolling axis of the plate) from the sheetmetal which underwent standard thermal treatment. The nominal diameter, gauge length and total length of these samples are respectively: 8.9, 50 and 108 mm. Test temperatures taken into account for these tests are room temperature, 300 °C and 600 °C. Tensile tests have been conducted as well according to ASTM E8/E8M on flat specimens extracted in l-T orientation from the plate which underwent recrystallization thermo-mechanical treatment. The thickness, gauge length and total length of these samples are respectively: 5, 25 and 130 mm. These tests have been carried out at room temperature only due to the limited amount and bended shape of the heavily cold worked and recrystallized plates (Fig. 1).

Such a bending is expected and due to the fact that RINA-CSM rolling machine is devoid of those "draw" devices with which larger scale production plants are equipped. The aim of testing tensile properties at room temperature only is just to have a term of comparison with the corresponding properties previously achieved on EUROFER 97/2 at the same temperature. 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 both at room temperature and at high temperature.

3. Results

3.1. Microstructural observations in order to detect PAGS

In Fig. 2 the microstructural observation aimed at identifying the PAGS on 3387 alloy is shown; In Table 3 the main parameters of the PAGS are reported. The mean PAGS results 7.3 μm, below the average value achieved after optimization for most of the martensitic steels taken into account in the past and for EUROFER 97/2 itself [9], in the range of 10 μm.

3.2. TEM microstructural observations in order to detect nature and size of the precipitates (extraction replica) and the recrystallized grain size (thin foils)

Information on chemical composition have been obtained by analysing around 250 particles by EDX (Energy Dispersive X-ray spectroscopy) microanalysis. Precipitate phases have been identified combining crystallographic analysis by SAD (Selected Area Diffraction) with EDX. Size distributions of total population of precipitates have been assessed on extraction replica with the aid of AIA (Automatic Image Analysis). The quantitative assessment of geometrical parameters was performed

Fig. 1. Waved shape of the heavily cold worked plate (alloy 3387).

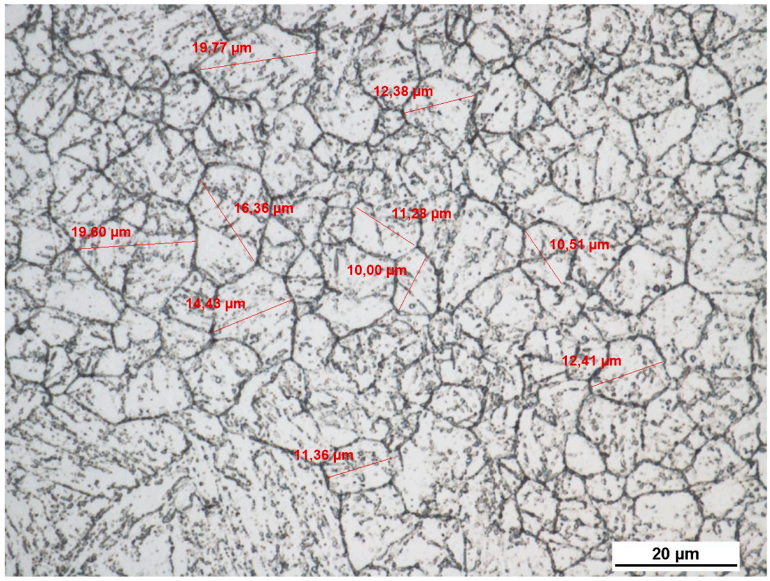

Fig. 2. LOM analysis of PAG (alloy 3387).

Table 3
Measurements of PAGS (alloy 3387).

Mean ASTM	St. Dev	Min ASTM	Max ASTM	Mean µm
11.2	0.2	11.0	11.5	7.3

by applying Feature Sizing software on several TEM images of extraction replica at different magnification (from 12,000 X up to 50,000 X) in order to achieve a relevant number of second phase particles (around 5000). For each TEM image, the grey level image file was converted to a binary image by using first image segmentation histogram (grey levels from 0 to 255) and then a particular grey level value was selected as a cut-off to form the binary image. The grey level range was chosen in order to obtain a good compromise between particle detection and separation between particles. Feature sizing was then applied on binary images after a selection of particle and field (frame) parameters. Feature sizing allows measuring several particle parameters. Precipitate size, D, has been assessed as the average between particle width and particle length. The geometrical parameters of the investigated steels have been assessed on >7000 particles. In Fig. 3 the identification of the precipitate nature is illustrated, by combining EDS analysis, to evaluate chemical composition, and SAD analysis to evaluate the crystallographic structure.

On the base of element repartition such as Cr, Fe, W, V, Ta, two main families of precipitates have been identified in both the steels (3387 and EUROFER97/2), with similar size and composition:

1. $M_{23}C_6$, Cr carbide, with minor W content

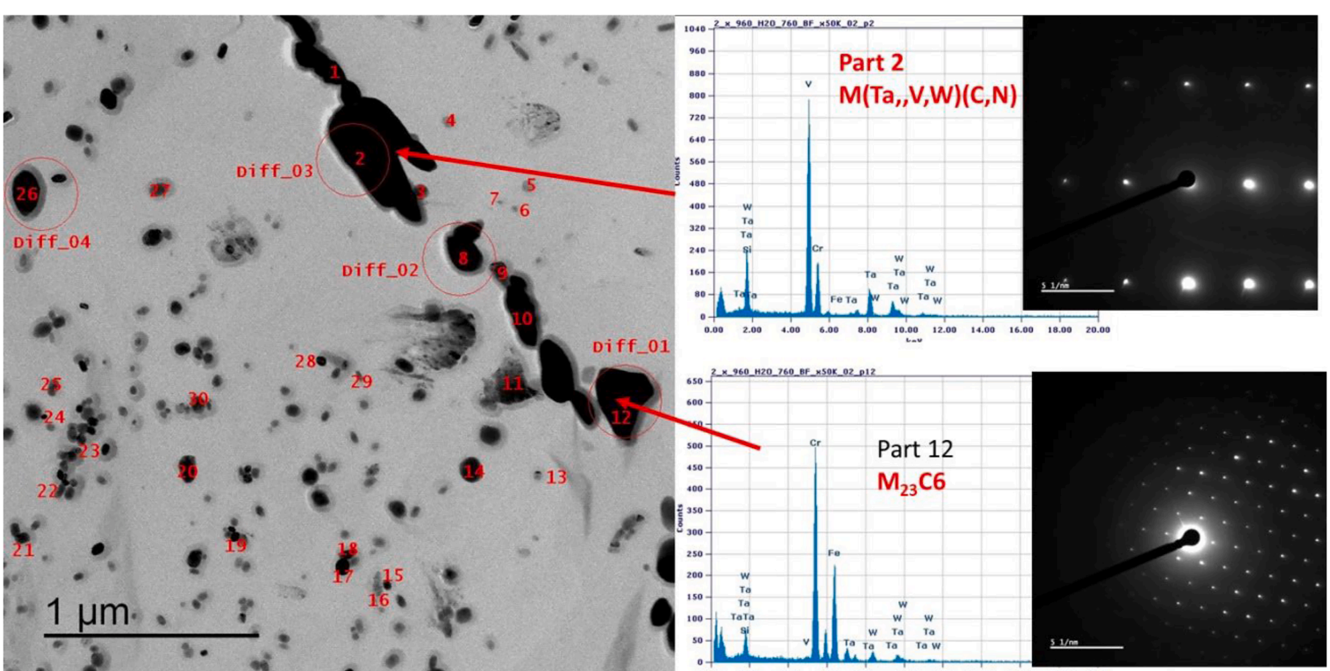

Fig. 3. Identification of precipitate nature combining EDS and SA analysis (alloy 3387).

2. MX carbo-nitride with low Cr content and, in some case, with minor W content.

In Table 4 the chemical composition, the mean size and the relative frequency number is reported: it can be observed that the mean size of MX precipitates is quite larger than that observed in other materials, being generally lower than 30–50 nm. In the table, for comparison, the measured values of standard EUROFER 97/2 are reported. Also, Cr-Fe carbides have larger size in the new heat, while their composition seems very similar to the previous detected. In 3387 alloy only one type of MX precipitate was detected, i.e. 66 %V-16 %Ta, and no rich Ta phase was present.

The compositions of the precipitates in the extraction replicas for the 3387 steel are presented in the ternary diagram of Fig. 4. The regions for Ta and V carbides, as well for Cr-Fe carbides, are clearly evidenced.

Precipitate size, D, has been assessed as the average between particle width and particle length. The geometrical parameters of the investigated steels have been assessed on >7000 particles. The quantitative information can be elaborated to compare the distribution of different steels. In Fig. 5 the precipitate diameter distribution measured on 3387, hot rolled and heat treated, is compared with that measured on Standard EUROFER 97/2: size distribution of 3387, compared to EUROFER 97/2, is shifted to higher mean size thus implying averagely coarser dimension of precipitates in the former respect to the latter. The double peak appearing in the EUROFER 97/2 distribution is associated to the two families of MX precipitates, Ta and V rich: the size distribution of 3387 shows only a single peak since only one type of MC precipitates has been detected. 3387 alloy and EUROFER 97/2 show the same values above ~170 nm.

The TEM analysis performed on thin foils (Fig. 6) confirms the microstructure already detected after application of the same treatment on EUROFER 97/2 [10]; the applied TMT (Thermo Mechanical Treatments) induces homogeneous recrystallization at the considered temperature. The grain size results in a range of 1 μm. A very low dislocation density can also be deduced.

The analysis on the distribution of precipitates has been carried out on the standard condition only for comparison with EUROFER 97/2; it hasn't been repeated after recrystallization as it is assumed that the low temperature of the final recrystallization tempering (550 °C) wouldn't affect the distribution of precipitates achieved in the previous tempering (760 °C) and the authors also got confirmation of this according to such analysis performed in the two condition (Standard and recrystallized) on another RAFM steel with similar chemical composition.

3.3. Impact tests (Charpy IsoV and KLST specimens)

The results of the impact tests on Charpy Iso-V samples, extracted from the plate which underwent standard treatment, are reported in the next plot (Fig. 7), both in terms of DBTT and FATT (Fracture Appearance Transition Temperature). The transition results located at −108 °C, well below the −80 °C achieved on EUROFER 97/2 [13]. Both the chemical composition changes, concerning the addition of Vanadium and Nitrogen, and the thermal treatment modifications, namely the double austenitization at 960° resulting in a PAGS in the range of 7 μm, are thought key features affecting this final outcome.

Table 4
Chemical composition of precipitate phases determined by TEM-EDS (%at).

		3387 alloy (960 °C/1 h/WC + 960 °C /1 h/WC + 760 °C/0.5 h)							
		Size	wt %					Frequency	
		(nm)	V	Cr	Fe	Ta	W	Nr	%
MX	75	66	13	<1	18	<1	52	79	
Cr-Fe	207	<1	59	31	0	9	14	21	
		EUROFER 97/2 (1020 °C/1 h/AC + 1020 °C /1 h/AC + 760 °C/1 h)							
		Size	wt (%)						Frequency
		(nm)	V	Cr	Fe	Ta	W	Nr	%
Cr-Fe	136	1	58	30	<1	9	63	68	
MX	Ta	21	5	5	2	79	5	20	21
	V	45	56	18	3	17	3	10	11

The results from the impact tests on KLST specimens are reported in the next plot (Fig. 8) and summarized in the next table (Table 5). The plot shows the comparison between double normalized-tempered plates and recrystallized ones. The two steels, namely EUROFER 97/2 and 3387, are taken into account in the comparison, in both thermal treatment conditions. The data from EUROFER 97/2, recalled here for comparison, have been already published in the past [10]. For the Standard conditions the transition is sharp, the DBTT results from the best fit (hyperbolic tangent function) of the experimental points and the value of the Standard Deviation is calculated automatically. For the recrystallized conditions instead, due to both the spread in the experimental points and the "smooth nature" of the transition, the same "best fit" approach cannot be followed in a straightforward way. Infact, applying the fitting criterion to the experimental points from recrystallization, the transition is interpreted as not completed and LSE is estimated lower than the values actually achieved experimentally, with a lower DBTT. Then, in order to be more conservative, it would be needed to add some "fake" points (for example −195, −200 °C, −205 °C), not achievable experimentally due to the limit temperature of liquid Nitrogen at −196 °C, in addition to the actually achieved experimental ones (−188 °C), in order to force the LSE, assuming the LSE to be the same at −188, −195, −200 °C, −205 °C. By this "artificial" (but somehow also conservative) procedure the DBTT results −160 °C, according to the fitting function. The value of the DBTT achieved this way from the fitting is confirmed by what can be observed directly in the plot (Fig. 8), assessing the DBTT at the left border of a sort of "plateau", occurring between −140 and −160 °C relying just on the experimental points themselves. This plateau corresponds to half of the energy range between LSE and USE. However the DBTT of the recrystallized alloys, due to the smoother nature of the transition combined to the higher spread of experimental points, is itself, for its intrinsic nature, affected by more relevant uncertainty. In Table 5 the DBTT value is indeed defined as "approximate" for this condition.

The comparison between KLST transition curves, associated to the observed microstructures, shows that:

- The new alloy (3387) exhibits an improved DBTT compared to EUROFER97/2, both after standard and recrystallization treatment. Namely the new steel shows higher residual toughness at very low cryogenic temperature (−130 °C for the standard condition; −160 °C for the recrystallized condition);
- The USE of the 3387 alloy is markedly higher after standard treatment compared to EUROFER97/2;
- The recrystallization treatment results in a smoother DBTT with a lower value of the transition compared to the standard treatment for both the steels: EUROFER 97/2 and 3387; this nature of the curve seems strongly correlated to the average dimension of the grain; namely the lower the grain size the better the behaviour at very low cryogenic test temperature.

Such a change in the impact behaviour of martensitic steels, comparing standard treatment to recrystallization one, seems consistent with the reduction of the grain size at sub-micrometric scale, according to several references [14–16]. Compared with the coarse-grained standard steels, in the UFG (Ultrafine Grained) steels the USE (Upper Shelf Energy) is decreased and the LSE (Lower Shelf Energy) is enhanced. The decrease in grain size leads to a decrease in the DBTT. Moreover the transition curve doesn't show a sharp drop in the absorbed energy, but exhibits a more gradual decrease in energy from the USE region to the LSE region. The increased amount of grain boundaries in the UFG microstructure improves the resistance to crack propagation [14].

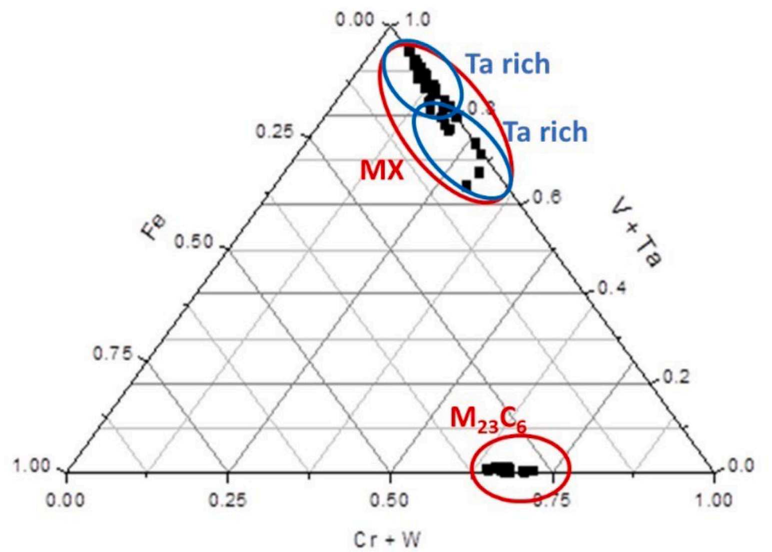

Fig. 4. Ternary diagram of precipitate compositions for 3387 alloy.

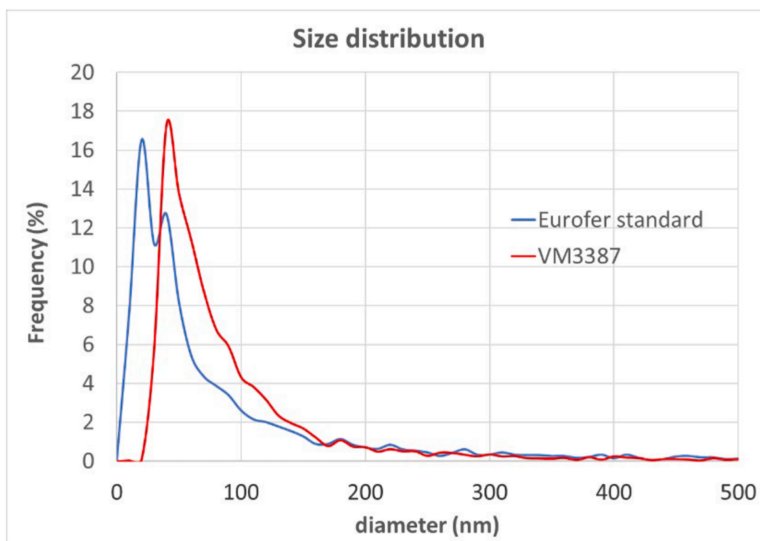

Fig. 5. Precipitate diameter distribution measured by AIA on extraction replicas (>7000 particles).

3.4. Tensile tests and hardness

The YS (Yield Strength) and UTS (Ultimate Tensile Strength) values achieved from the tensile tests are reported in the next plot (Fig. 9). The EUROFER 97/2 values to which these ones are compared come from [13,17] concerning standard treatment and from [10] concerning recrystallization treatment. The tensile properties of 3387 alloy result markedly enhanced after recrystallization compared to standard treatment; 150 MPa profit on the YS and 130 MPa on the UTS at room temperature. The enhanced tensile properties after recrystallization are consistent with the difference respect to the standard treatment previously achieved on EUROFER 97/2. The higher tensile properties after recrystallization are also consistent with the recorded values of hardness; 240 HV5 in the recrystallized condition and 195 HV5 after standard heat treatment. Whether these increased tensile properties after recrystallization depend more on Hall-Petch effects (1) related to reduced Grain Size or on residual dislocations density after cold working and 550 °C tempering has still to be demonstrated.

$$\sigma_y = \sigma_0 + \frac{K_y}{\sqrt{d}} \quad (1)$$

Where σ_y is the Yield Strength, σ_0 is a constant specific to each material representing the threshold stress for dislocation motion, K_y is the strengthening coefficient (a constant specific to each material) and *d* is the average grain diameter.

Nevertheless the tensile strength of 3387 alloy results lower (80 Mpa less on the Yield Strength) than EUROFER 97/2, both in the standard and recrystallized condition. It can be deduced that the modifications to the EUROFER 97/2 chemical composition as well as the coarser distribution of precipitates achieved in 3387 alloy can be correlated to this decay in tensile properties.

4. Conclusions

The new heat 3387, which involves chemical composition changes respect to EUROFER 97/2, particularly in terms of Vanadium, Nitrogen and Manganese content, was investigated both after Standard HT (hot rolled and heat treated) and with the innovative TMT (cold rolling and

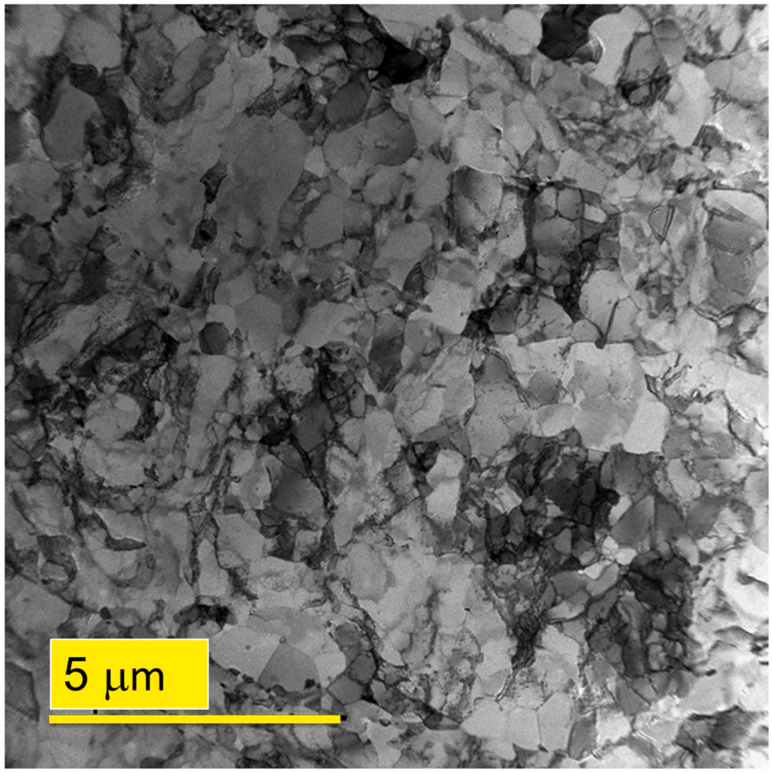

Fig. 6. TEM analysis (thin foils) of 3387 alloy, 50 % cold rolled and tempered (550 °C x 1 h) to achieve recrystallization.

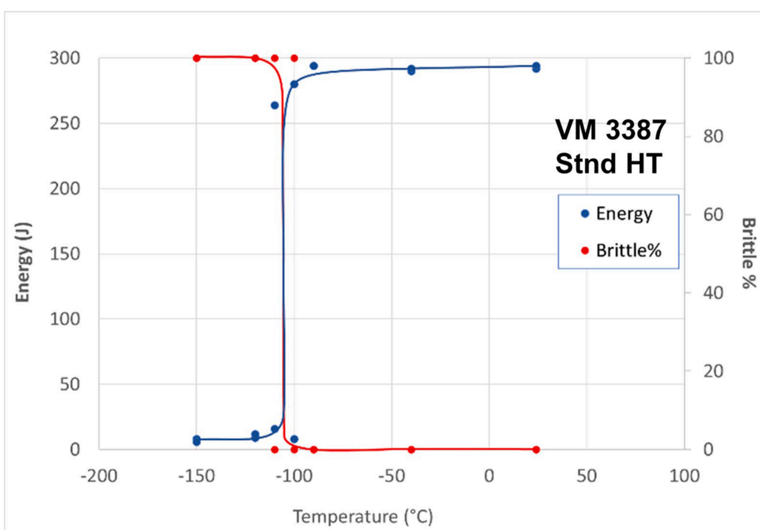

Fig. 7. Charpy Iso-V test results after standard treatment on 3387 plate.

recrystallization treatment). The main conclusions are the following:

- The double austenitization treatment and the control of the chemistry allowed obtaining on hot forged material a very fine microstructure compared to other materials previously investigated, with PAG mean size around 7 μm, with a recovered martensitic microstructure, characterized by a low dislocation density and a relatively low hardness (195 HV5);
- The DBTT after standard HT is −108 °C by standard ISO-V specimens (−140 °C by KLST ones), lower than that observed for standard EUROFER 97/2, which is around −80 °C (−117 °C by KLST);
- The cold worked material shows a fully recrystallized microstructure with grain size lower than 1 μm, and hardness of 240 HV5; the tensile properties of 3387 alloy result markedly enhanced after recrystallization compared to standard treatment; 150 MPa profit on the YS and 130 MPa on the UTS at room temperature;
- The recrystallized 3387 alloy exhibits very high residual toughness at very low temperature (DBTT approx. = −160 °C), with a USE range comparable to EUROFER 97/2;
- The tensile properties (YS and UTS) of 3387 alloy are lower than EUROFER 97/2 (both in the standard and recrystallized condition); about 80 MPa decay on the YS after standard treatment;

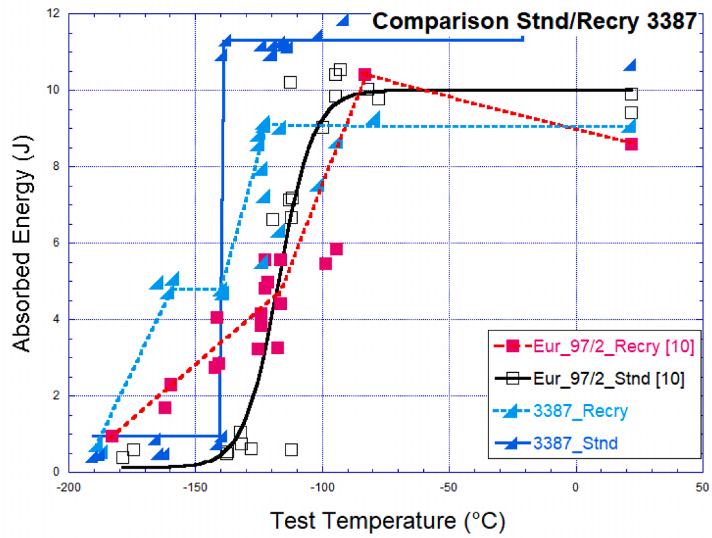

Fig. 8. KLST test results after standard and recrystallization treatment on 3387 plate; EUROFER 97/2 data from literature [10].

Table 5
Mean value and standard deviation of the results of the impact tests on KLST specimens; EUROFER 97/2 data taken from [10].

Status of steels	Mean value of the DBTT (°C)	Standard deviation (°C)	USE (J)	LSE (J)
Standard HT EUROFER 97/2	−117	2	9.98	0.48
Recrystallized/50 % c. w./550 °C EUROFER 97/2	−126 (approximate)	–	9.5	1
Standard HT 3387	−139	1.5	11.2	0.6
Recrystallized/50 % c. w./550 °C 3387	−160 (approximate)	–	9	0.6

- The new steel shows a coarser precipitate distribution compared to EUROFER 97/2. The coarser distribution of precipitates recorded on 3387 alloy could be correlated to the decay in tensile properties but not to the improved impact properties; namely the toughness of the steel appears more sensitive to grain size rather than to precipitate distribution.

To summarize we can say that the strategies adopted at the alloy design stage prove very effective in improving impact properties, but at the expense of a marked decay in tensile properties. This suggests that Carbon (whose content is higher in EUROFER 97/2) is much more effective than Nitrogen (whose content is increased in 3387 alloy) concerning the precipitation of those compounds (carbides more than nitrides) whose amount is strongly connected to tensile properties. However, for the WCLL-BB scenario, due to the foreseen “low

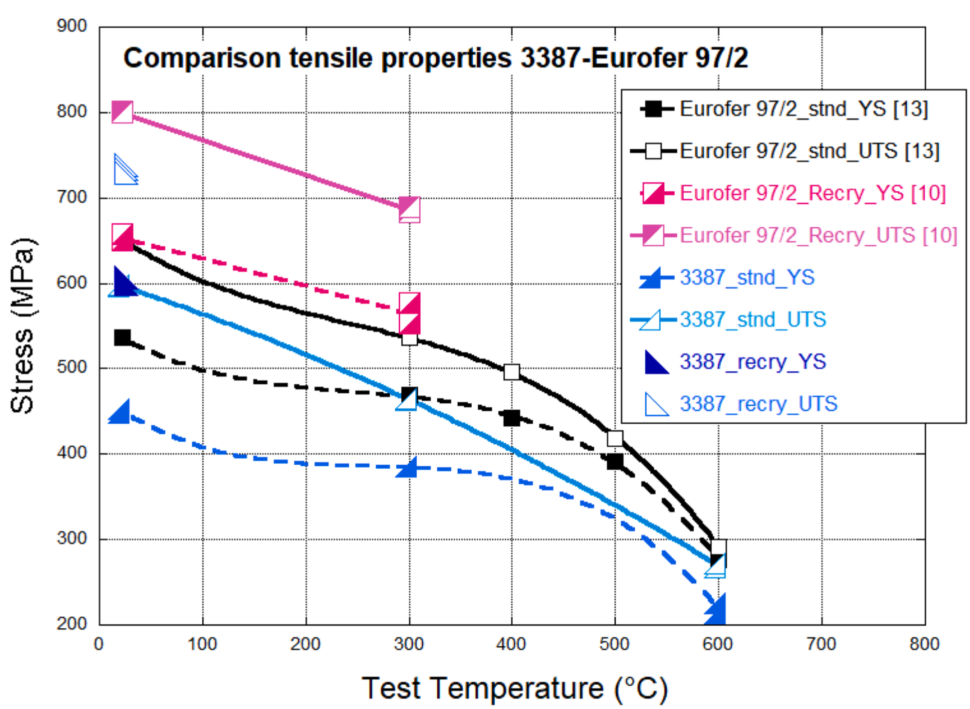

Fig. 9. Tensile properties of 3387 alloy; EUROFER 97/2 data from literature [13,10].

temperature" operation, the requirements concerning impact properties are considered much more demanding than those on high temperature tensile properties [18], and then the authors have taken into more consideration, and have devoted more of their efforts and resources, to the improvement of the former rather than the latter, at this stage. It is also well known that on martensitic steels the improvement of impact properties is generally accompanied by a decay in tensile properties; for example the tensile properties of 3387 alloy could be improved by simply decreasing the tempering temperature in the standard treatment, but this would likely result in a negative effect on impact properties. In any case the strengthening contribution of secondary MX phase at the tempering stage appears more negligible than that of primary MX phase at the normalization stage. Comparing 3387 to 2991 [8] infact, the strength of 3387 alloy is much inferior to 2991 alloy, and this can be attributed to the coarsening of primary phase particles provided that the normalization temperature of 3387 alloy has been kept markedly lower (960 °C) than the one adopted for 2991 alloy (1080 °C) which was meant to provide better solubilization of carbides at the expense of PAGS. The tempering temperature is the same (760 °C) for the two alloys and then coarsening of secondary MX phase should have minor influence on the achieved tensile strength. The experimental results seem to imply that coarsened MX phase is harmful to the improvement of tensile properties, even though the lower double austenitization temperature as well as the recrystallization have contributed to enhance the toughness of this alloy.

Another issue concerns the possibility of welding such steels, since traditional high temperature welding techniques would inevitably destroy such microstructures with a consequent loss of all its benefits for the component. It's known that some alternative low temperature welding techniques (soldering, brazing, friction welding, explosion welding, laser welding) do actually exist, but the authors hadn't the possibility, up to know, to carry out a systematic survey on the existing low-temperature welding techniques in order to explore this field at a deeper level and understand whether such welding technologies could be realistically upscaled to the thicknesses and complex shapes of the sheetmetals of the breeding blanket modules.

A general remark in the end is that the refined microstructure achieved first of all after recrystallization, but, to a minor extent, also after double normalization in the standard treatment, if compared to the PAGS previously achieved on EUROFER 97/2 and other martensitic alloys, is expected to be more tolerant to the neutron irradiation embrittlement. Another general remark is that the residual ductility at the very low cryogenic temperatures, achieved with this new 3387 alloy in the recrystallized state, is thought to represent a key feature in limiting the irradiation embrittlement, provided that a shift in the DBTT occurs under irradiation. Even if, under irradiation, we cannot assume that the curve will be shifted parallel and equal in the Upper and Lower Shelf Energies, this retained ductility towards the Lower Shelf regime is however sympthomatic of an alloy less prone to brittle failure, and the same behaviour is expected also under irradiation. On the one hand, concerning the standard treatment, the sharp DBTT is shifted towards the lower cryogenic temperatures compared to EUROFER 97/2. On the other hand, concerning the recrystallization treatment, according to the literature sources reported before, the smaller the grain, the smoother the transition from ductile to brittle regime. Therefore the smoother transition in the recrystallized condition is itself a clue of a deeply refined microstructure and the reduced grain size has to be considered a benefit under irradiation both from the point of view of the displacement cascade and in light of the Helium absorption; the increased fraction of grain boundaries acting as sinks for point defects and as absorption sites for Helium is expected to result in a delayed onset of neutron irradiation embrittlement.

CRediT authorship contribution statement

Carlo Cristalli: Writing – review & editing, Data curation, Conceptualization. **Luciano Bozzetto:** Resources, Data curation. **Alessandro Colaneri:** Resources, Data curation. **Roberto Sorci:** Resources, Data curation. **Oriana Tassa:** Data curation, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Carlo Cristalli reports financial support was provided by European Consortium for the Development of Fusion Energy. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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