Material: Ferritic Steel: F82H-mod.
Property: Temperature (˚C) versus Energy (J)
Condition: heat treated
Data: Experimental

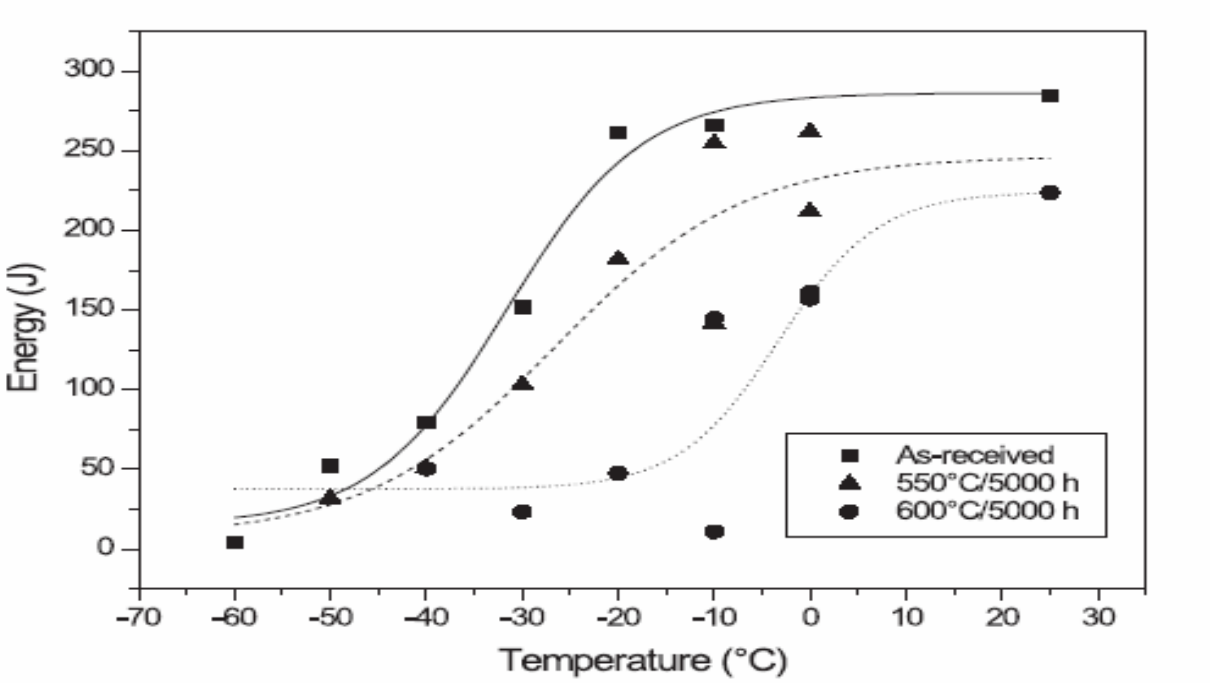


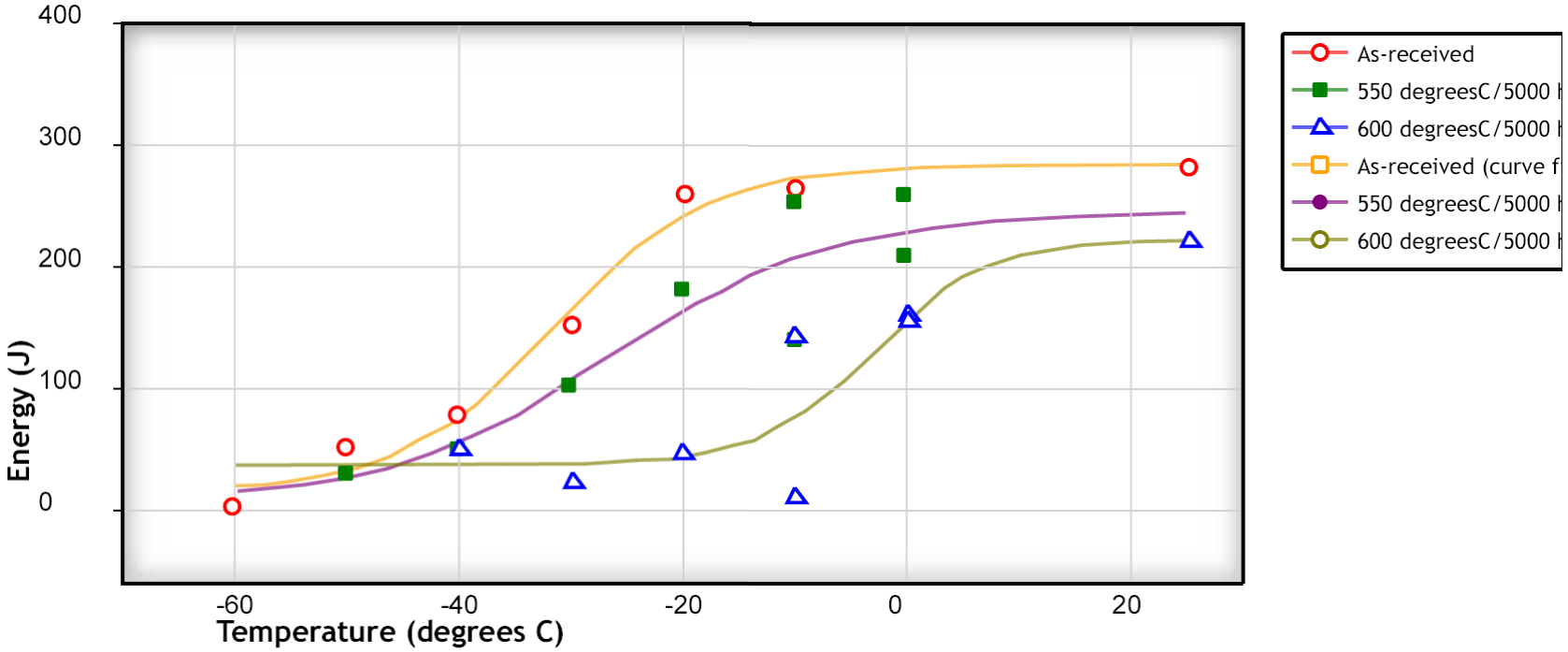
Fig. 1. Impact properties of F82H-modified steel.

Source:
Journal of Nuclear Materials, 283-287, 2000, 662-666

Title of paper (or report) this figure appeared in:
Chemical segregation behavior under thermal aging of the low-activation F82H-modified steel

Author of paper or graph:
J. Lapena, M. Garcia-Mazario, P. Fernández, A. M. Lancha

Caption:
Impact properties of F82H-modified steel.



Impact properties of F82H-modified steel.

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Plot Format:

Y-Scale: ◉ linear ○ log ○ ln

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Chemical segregation behavior under thermal aging of the low-activation F82H-modified steel

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Abstract

In this work, thermal aging of the low-activation F82H-modified steel has been performed at temperatures in the range 300–600°C during periods up to 5000 h. A detailed mechanical and microstructural characterization has been carried out in the aged materials, as well as in the as-received state material for reference. Auger electron spectroscopy (AES) analysis has been performed for these materials to study the microchemistry at the grain boundaries. The results show a decrease of the impact properties after aging at 600°C that has been related to the precipitation of the Laves phase. Auger analyses show chromium enrichment and iron depletion at grain boundaries in all material conditions. In addition, sulphur and tungsten have been observed by this technique at grain boundaries, their presence and distribution being dependent on the material state.

1. Introduction

Low-activation ferritic/martensitic steels, such as the F82H steel, are being considered for use as structural materials in future fusion energy systems. In these applications, they must not only withstand radiation damage, but also high temperatures for long periods of time. Therefore, an exhaustive knowledge of the mechanical and microstructural characteristics of these materials aged for a long time at high temperatures is necessary in order to understand their subsequent behavior under irradiation.

In this work, thermal aging of the low-activation F82H-modified steel has been performed at temperatures in the range 300–600°C during periods of up to 5000 h with the objective of gaining some insights into the long thermal aging embrittlement mechanisms of this steel. A detailed mechanical and microstructural characterization has been carried out in the aged materials, as well as in the as-received state material for reference. Auger electron spectroscopy (AES) analysis has been performed for these materials to study the microchemistry at the grain boundaries in order to determine the segregation of major and minor elements and impurities.

2. Experimental

2.1. Material

The material used in this study was a reduced activation ferritic/martensitic steel F82H-modified (7.65Cr, 2.1W, 0.100C, 0.16Mn, 0.14V, 0.003S, 0.02Ta wt.%, balance Fe). This steel was supplied as plates in the normalized (1040°C/37 min) and tempered (750°C/1h/air cooled) conditions. The alloy was thermally aged at 300°C, 400°C, 500°C, 550°C and 600°C for periods of 500, 1000 and 5000 h.

2.2. Microscopy

The microstructural characterization of as-received and aged material was carried out by optical and scanning electron microscopy (SEM). In addition, specimens from the as-received condition and aged at 600°C for 5000 h were studied by transmission electron microscopy (TEM) on a 200 KV JEOL instrument. In these cases, thin foils and carbon extraction replicas were prepared.

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Phase extraction was performed in all material conditions by anodic dissolution of the matrix. The extracted residues were analyzed by X-ray diffraction and EDX (SEM).

2.3. Mechanical testing

Tensile specimens from as-received and aged material (300°C, 400°C and 500°C for 500, 1000 and 5000 h) were tested at temperatures of 300°C, 400°C and 500°C. Creep specimens from as-received and aged material (550°C, 600°C during 5000 h) were tested in air temperatures in the range of 450–650°C and stresses in the range of 80–320 MPa. To determine the impact properties of F82H, Charpy impact tests were conducted on as-received and thermally aged material (550°C and 650°C for 5000 h) according to ASTM E23. After impact testing, the fracture surfaces were examined by SEM.

2.4. Auger electron spectroscopy (AES)

At least two notched cylindrical samples were studied per condition. Samples were cathodically charged with hydrogen, and then they were fractured by tension at a deformation rate of 1 μm/s inside a PHI660 Auger chamber at a pressure of 10^{-9} Torr. Spot analyses were performed on grain boundary facets and on ductile fracture for comparison. The atomic concentrations were calculated according to [1,2] and were normalized to 100%. Oxygen and carbon were not included in the normalization because they are subject to errors due to environmental contamination during long examinations. Sputtering depth profiling was performed using a 2.0 keV argon ion flux, the etching rate calibration being achieved with a Ta_2O_5 thin film of known thickness.

3. Results and discussion

3.1. Microstructure

The microstructure of the material in the as-received condition was fully martensitic, with lath-shaped subgrains of ~1 μm wide, containing large amounts of carbides precipitated preferentially at the prior austenitic grain boundaries and at the lath boundaries, but also in the bulk of the martensite lathes. The carbide size was in the range 0.05–0.3 μm. The average grain size was ASTM 4.5–5.5. After the different aging treatments, no significant differences were observed optically and by SEM in the microstructure of the material. A stable microstructure of the F82H-modified steel under aging has also been observed by other authors [3].

The results of the phase extraction tests showed that the amount of extracted residues increased on the aged material with respect to the as-received state (1.5% extracted residue), slightly for 400–550°C (~2.6%) and appreciably for 600°C (4.1%). The EDX analyses performed on these residues indicate the presence of Fe, Cr, W and V, their concentrations in the as-received condition being in accordance with literature results [4]. A slight increase of W (5 at.% in as-received) were seen for the aging temperatures of 400–550°C (~7 at.%), this increase being significant for the temperature of 600°C (14 at.%).

By X-ray diffraction, $M_{23}C_6$ carbides were identified in the as-received and aged materials. The X-ray diffraction pattern corresponding to the thermally aged material at 600°C also showed weak peaks of Laves phase that were not detected in the other material conditions. The TEM studies performed on aged material at 600°C also confirmed the presence of Laves phase (Fe_2W) in this condition while, in the as-received material, only $M_{23}C_6$ carbides were seen. No correlation between Laves phase and pre-austenite grains was observed.

The precipitation of Laves phase under thermal aging at 600°C/5000 h is in accordance with the time–temperature–precipitation (TTP) diagrams for Laves phase in this material [5]. Similar results were obtained by Tamura et al. [6] in work performed on the F82H, for which an increase of extracted precipitates was also observed at 600°C due to the precipitation of Laves phase.

3.2. Mechanical properties

Tensile properties of as-received F82H showed adequate strength and ductility levels which were comparable with other low-activation ferritic/martensitic steels [7]. No degradation of properties was observed in aged material. These results were in agreement with the high stability of the microstructure observed and with the hardness values, 210HV10, measured in all cases. The behavior of F82H modified in the creep tests showed that for the same load and temperature test the three materials conditions tested had the same creep properties.

Charpy tests were conducted on each material state studied over a range of temperatures in order to establish full DBTT curves, Fig. 1. Note that, in contrast to tensile and creep properties, impact behavior seems to be sensitive to the metallurgical condition of the material. The effects are more important on aged material at 600°C, which exhibits a DBTT shift of about 18 J and a decrease in USE of approximately 45 J, both compared with the as-received state. The SEM examination revealed that the brittle fracture in the lower shelf region was produced transgranularly by quasi-cleavage in the martensite phase. No grain boundaries were observed.

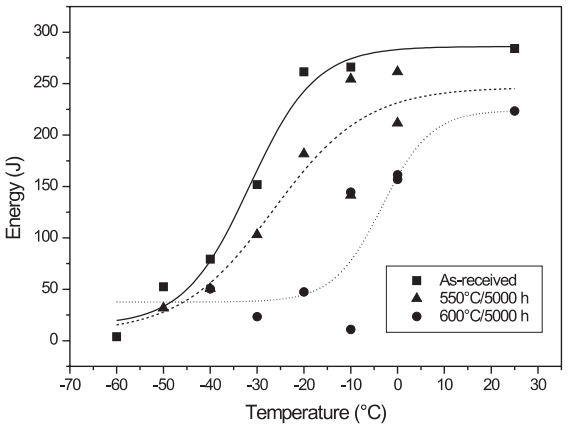

Fig. 1. Impact properties of F82H-modified steel.

Therefore, based on the microstructural examination by TEM, and on the amount and composition of the residue of the aged material extracted at 600°C/5000 h, it can be stated that the Laves phase precipitation is the dominant factor for the decrease of the impact properties of F82 steel. Similar results of decrease in toughness on 9%Cr–2%Mo and 8%Cr–2%W have been reported by others authors to be closely related to the precipitation of Laves phase [6,8].

3.3. Grain boundary microchemistry

In the as-received condition, three samples were fractured and a total of 170 analyses were performed at the intergranular facets. Fig. 2 shows a photograph of one zone which was analyzed and the histograms of iron, chromium and sulphur. As can be seen, iron concentration is lower in the intergranular (78 ± 7 at.%) than in the ductile areas (90 ± 2 at.%), while chromium presents an inverse trend, its concentrations being clearly higher at the intergranular zones (16 ± 6 at.%) than in the ductile ones (9 ± 1 at.%). Relating to sulphur, the histograms show this element clearly in a higher percentage of analyses and with higher concentrations at the intergranular areas than in the ductile ones. No relation of sulphur with manganese or other metals forming particles in the intergranular areas has been observed. Therefore, it can be said that sulphur is present at grain boundaries but heterogeneously distributed. By sputtering, the presence of sulphur and chromium at the grain boundaries was determined up to a depth of 20 nm.

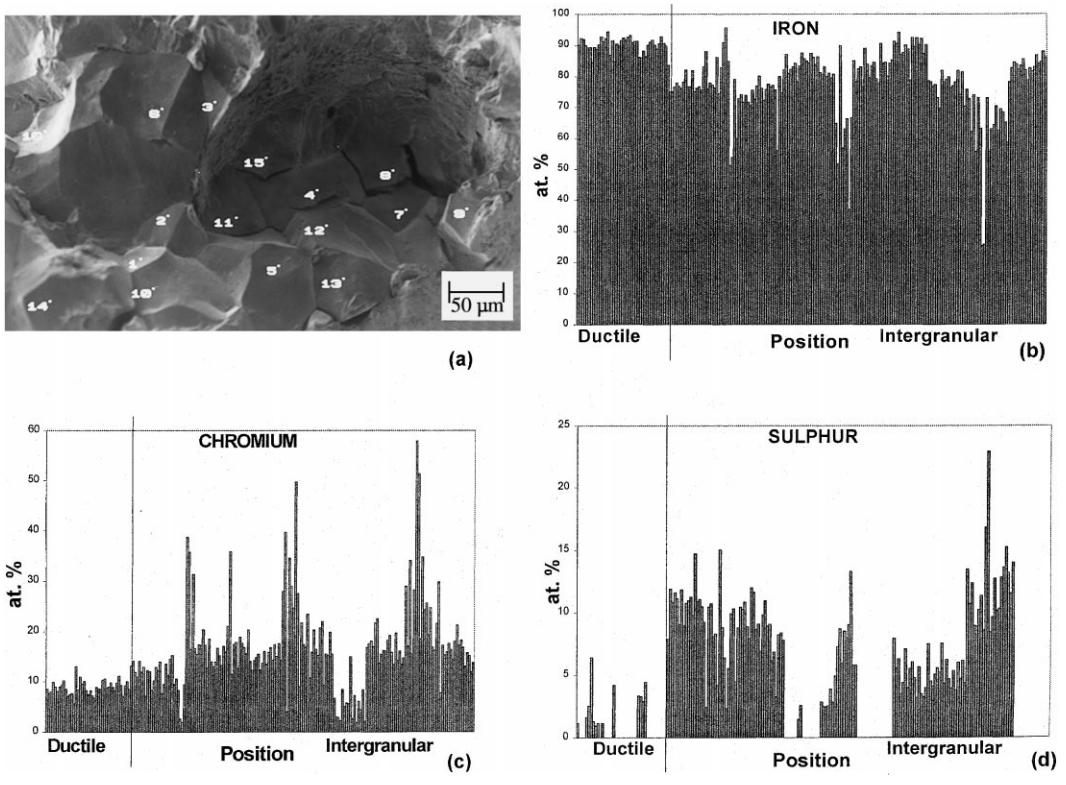

Fig. 2. Auger analyses in the as-received condition: (a) SEM image of a fracture surface, (b), (c) and (d) histograms showing the atomic concentrations versus position (intergranular or ductile) for iron, chromium and sulphur, respectively.

Chromium enrichment and iron depletion at grain boundaries of F82H steel in the as-received state have been reported by other authors [9]. Unfortunately, no information is available about grain boundary sulphur segregation in this steel. The presence of chromium in pre-austenite grains is believed to strengthen the boundaries [9,10]. In the opposite sense, the chromium depletion of grain boundaries enhances the intergranular fracture of ferritic steels [9,11].

The aging treatments at 400°C, 500°C and 600°C for 5000 h showed the following results. At 400°C, a very small percentage of intergranular fracture was obtained and, thus, few intergranular facets were analyzed, showing great scatter. In spite of this, the material in this condition also showed clear chromium enrichment and iron depletion at grain boundaries. The aging treatments at 500°C and 600°C did not change this trend.

Relating to sulphur, its segregation is dependent on temperature, Fig. 3. An arrow indicates the average value (calculated considering only the analyses with sulphur). A clear and interesting trend can be observed. In the as-received state, as commented on previously, sulphur is heterogeneously distributed, with ~27% of the grains analyzed without sulphur. This could be a consequence of a micro-segregation process during manufacturing. At 500°C, the majority of the grains were free of sulphur, showing only a very small amount of sulphur in the remaining grains and up to a depth $\leqslant 10$ nm. After the heat treatment at 600°C, practically all the grains showed sulphur, in concentrations slightly higher than in the as-received state. These results seem to be a bit surprising taking into account that a higher percentage of intergranular fracture was obtained at 500°C (~42%) than in the as-received/600°C conditions (~18%). At 600°C, four sputtering depth profiles showed variable sulphur depths ranging from 0.4 to 30 nm. Therefore, a migration process towards the matrix is believed to occur under aging at 500°C, while, at 600°C, sulphur seems to segregate to interfaces in a homogeneous way.

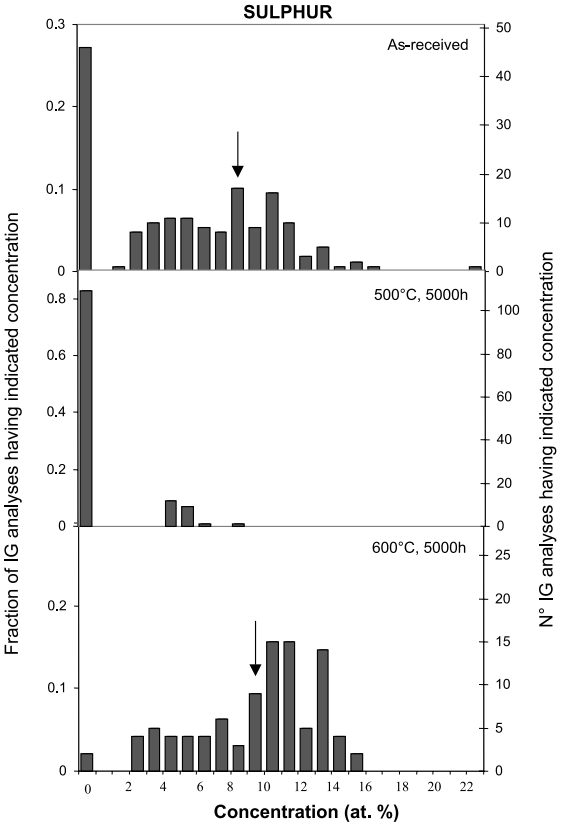

Fig. 3. Histograms of the intergranular surface concentrations of sulphur.

On the other hand, it is worth mentioning that small amounts of phosphorus were detected in 11% of the grains analyzed at 500°C. No sign of phosphorus was detected in the as-received state or in the rest of the aged conditions.

It is well known that the intergranular segregation of sulphur and phosphorus weakens cohesion at grain boundaries in ferritic/martensitic alloys. Moreover, it has been recognized that the embrittlement potency of sulphur is greater than that of phosphorus [12]. However, in this study, the significant shift in the DBTT after aging at 600°C, without the observation of intergranular fracture, seems to indicate that sulphur segregation to prior austenite grain boundaries is not directly responsible for decreased toughness. In spite of this, the relevance of the grain boundary results obtained in this work must be noted, taking into account the fact that more severe conditions operate in welded regions of these materials and much intergranular failure has been seen in other ferritic steels in these locations.

Tungsten was also detected at grain boundaries in aged materials. In the as-received condition, no tungsten was observed in any of the 170 points analyzed. However, after aging at 500°C, this element was detected in almost half of the intergranular analyses, Fig. 4. When the aging temperature increases to 600°C, the percentage of intergranular points without tungsten rises close to 70%, presenting the rest of the analyses values of this element slightly higher than for 500°C. By four sputtering depth profiles, the presence of tungsten was determined up to depths of 0.4, 2.5, 5 and 50 nm for the heat treatment at 500°C, while, for 600°C, the depths observed for its disappearance were 10 nm in one profile and in the range of 120–200 nm for the remaining profiles.

These results seem to be in accordance with the EDX analyses of the extracted residues, which also showed higher values of tungsten after aging, and they indicate that, after aging, grain boundaries are being enriched in tungsten. The observed depths seem to indicate that, at

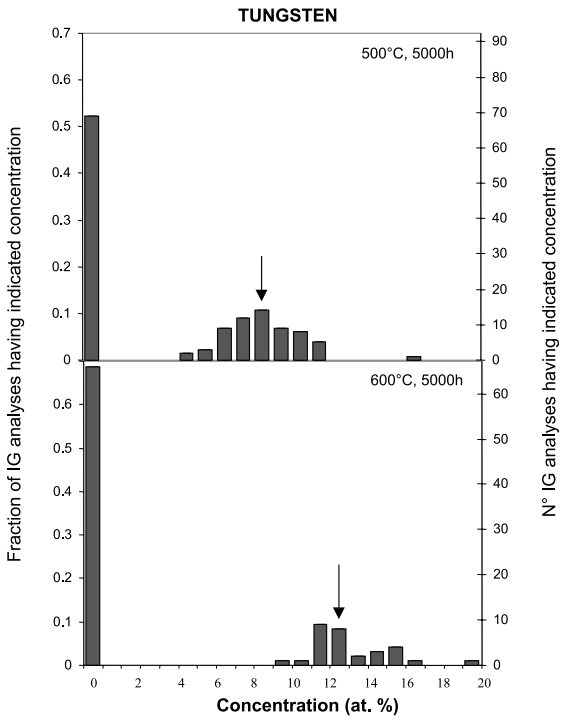

Fig. 4. Histograms of the intergranular surface concentrations of tungsten.

500°C, there is mainly segregated tungsten while, at 600°C, the tungsten is present probably in the form of particles. This would mean that tungsten segregates to interfaces but, when its concentration reaches a critical value, precipitation occurs. The tungsten at grain boundaries would be associated with the presence of Laves phase, but this point has to be confirmed before correlating it with the reduction of impact strength. Some authors [13] have stated the formation of Laves phase distributed as thin films along grain and subgrain boundaries at ~500°C in a 9Cr–1MoVNb steel, while Laves phase is formed as coarser particles at temperatures $\geqslant$ 600°C. SEM examination of the intergranular facets at high magnification and complementary studies of grain boundary microchemistry and material microstructure by TEM/EDS are still in progress for a better understanding of the chemical behavior of tungsten at grain boundaries and its correlation with the impact properties.

4. Conclusions

- A decrease of the impact properties of F82 steel has been observed after aging at 600°C for 5000 h that has been related with the precipitation of Laves phase. Laves phase has been identified by TEM and X-ray diffraction on extracted residues.
- Chromium enrichment and iron depletion at grain boundaries has been detected in the as-received state. This trend is not affected by temperature in the range studied.
- Sulphur has been seen at grain boundaries distributed heterogeneously in the as-received condition. At 500°C, sulphur was not detected at grain boundaries while, at 600°C, it seems to segregate homogeneously to interfaces.
- No tungsten was detected by AES at the grain boundaries of the as-received F82 steel. However, after aging at 500°C and 600°C, tungsten enrichment at intergranular areas was clearly observed by this technique.

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