MECHANICAL CHARACTERISTICS OF 5754-NET-O ALUMINUM ALLOY IRRADIATED UP TO HIGH FLUENCES : NEUTRON SPECTRUM AND TEMPERATURE EFFECTS

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ABSTRACT

The 5000 and 6000 series aluminum alloys are extensively used in research reactors. To validate the choice of the suitable Al-alloys for the core components and the experimental devices for the conception of the Jules Horowitz Reactor (RJH), a characterization program of some highly irradiated components was performed. In this paper, we focalise on the 5754-NET alloy in the annealed temper (O treatment) which was irradiated under different neutron spectrum, in Osiris and Orphée reactors.

Tensile test results on this alloy irradiated for 30 years in OSIRIS are presented. The temperature and fluence effects are discussed.

Tensile test results on the 5754-NET alloy irradiated for 15 years in ORPHEE in a cold neutron flux (cold source shell) are provided and compared with results obtained after irradiation with a harder neutron spectrum in Osiris. Neutron calculations in the ORPHEE cold source shell show that the total flux is hardly reduced by the presence of the cold source, but the Si production rate is increased by a mean factor of 1.53. This Si production rate is dependent on the circumferential position relative to the reactor core. The comparison with the data on the alloy irradiated in Osiris shows that the total elongation after irradiation is directly related to the Si content created under neutron flux, whereas the mechanical strength is hardly affected by the high Si production rate due to the cold neutron flux. The neutron spectrum effect is discussed and compared to published results.

Microhardness measurements on the TIG-welding joins show that, on the unirradiated sample, the melted zone is slightly harder than the base metal. This difference is smoothed at high neutron fluence.

All these results contribute to a better knowledge of aluminium alloys properties under irradiation in research reactors. This topic is very important for better risk and safety analyses of this type of reactors.

1. INTRODUCTION

The low operating temperatures in the research reactors allow an extensive use of aluminium alloys for the conception of the reactor components (vessel, thimbles) and the experimental devices. Their good formability and their reduced cost make them attractive.

In the frame of the Jules Horowitz reactor conception, a characterization program of some highly irradiated components was performed to select the suitable Al-alloys for the core components and the experimental devices.

The 6061 Al-alloy is frequently used in the T6 temper for pressure vessels and in lots of components where a high mechanical strength is required. It has indeed a high mechanical strength, and a relative good stability under neutron flux. But the welding processes on the 6000 series heat treatable alloys induce an overheating of the melted zone and a mechanical strength loss due to the precipitation hardening annihilation. Aluminum alloys of the 5000 series are therefore generally preferred for reactor components entailing welding zones, when the strength requirements lie under those of the stronger precipitation-hardened 6000 series alloys. The 5000 series alloys in the annealed conditions are commonly used in European reactors for the external shell of irradiation loops, and the thimbles. The 5754-NET alloy, which is a modified specification of the 5754 ASTM grade aimed to reduce intergranular corrosion and to limit neutron absorption and activation, has been extensively used in the annealed condition in French research reactors. The TIG-welding process on this alloy does not alter the mechanical characteristics because this alloy is a solid solution strengthened by magnesium.

The tensile properties measured on the 5754-NET-O alloy irradiated for 30 years in Osiris with a neutron spectrum index close to one are presented, and discussed in terms of temperature and fluence effects.

The presence of cold sources has a thermalizing effect on the neutron spectrum. This spectrum effect on the mechanical characteristics of the irradiated 5754-NET-O alloy is investigated: tensile test results obtained on a cold source shell in Orphée are provided, and neutron calculations are presented. This post-irradiation program shows that the vicinity of a cold source should be taken into account in the life extent evaluation of some components (thimble neutron guides, cold source shells). These results have an impact on the safety analyses of research reactors.

The TIG-welding joins are often located in zones which render impossible the sampling for mechanical tests. The irradiation effect on the TIG welding zones in the 5754-NET-O alloy is estimated on the basis of micro-hardness measurements.

2. EXPERIMENTAL CONDITIONS

2.1. OSIRIS lattice structure

The Osiris lattice structure is the component which contains the core. It was already described in the figure 1 of [1]. The structure looks like a bottle rack in which are inserted the

fuel elements, being cooled by light water circulating upward. The water enters the core at a mean temperature of 38°C and gets out at around 47°C, and the irradiation temperature of the alloy is estimated to be within 50-55°C.

This structure was irradiated for 30 years, under a maximal conventional thermal neutron flux of $2,0x10^{14}$ n.cm⁻².s-1 (E=0.0254 eV) and a maximal fast neutron flux of $2,1x10^{14}$ n.cm⁻².s⁻¹ (E>1 MeV) in the maximal flux plane of the characterized cell. In this plane, the ratio of the thermal (E=0.0254 eV) to the fast (E>1 MeV) neutron flux is 0.95. At the top of the structure over the fuel elements, this ratio is about 8.

The 5754-NET alloy contains 2.75% Mg- 0.29%Fe- 0.19%Cr- 0.31%Mn - 0.08%Si.

The structure was machined in a forged block and the cell walls have a thickness of 3 mm. Tensile specimens were machined vertically at three different levels of one cell, chosen for its high fluence.

The thermal neutron fluence was deduced at each level from silicon content measurements after and before irradiation, by laser ablation optical emission spectroscopy technique (LA-OES). As the cross section for neutron absorption on ²⁷Al presents resonances in the epithermal energies range, the epithermal flux contributes significantly to the silicon production. The integrated thermal fluence in the [0; 0,625 eV] interval is therefore pertinent to express the silicon production damage on the alloy. This integrated fluence (Table 1) was estimated by neutron calculations, taking into account a simplified power history.

In table 1 the conventional fluence is also given. It is defined as the fluence at E= 0.0254 eV which would produce the measured generated silicon content, if all the neutrons had this energy. It is evaluated using the simple correlation: a thermal fluence of 10^{22} n/cm² (E= 0.0254 eV) produces 0.236 weight% silicon. This fluence is of interest in order to compare different components irradiated in different neutron spectrum conditions but to the same generated silicon content.

| Piece reference | Level | %Si generated | Thermal fluence $(E \le 0.625 \text{ eV})$ | Thermal fluence $(E=0.0254 \text{ eV})$ |
|--------------------|---------|---------------|--|---|
| | Law | | (1, 0, 023, 0, 1) | (10,020+0.1) |
| AI- | | 0.072 | 5.9/X10 ⁻⁴ | 5.05X10- |
| | fluence | | | |
| A1+ | Low | 0.197 | 10.8×10^{21} | 8.35x10 ²¹ |
| | fluence | | | |
| H12 | Mid | 1.12 | 6.18×10^{22} | 4.74×10^{22} |
| | fluence | | | |
| H31 | High | 2.12 | 11.7×10^{22} | 8.98x10 ²² |
| | fluence | | | |

<u>Table 1</u>

2.2. ORPHEE cold source shell

This component is a vertical closed tube under vacuum, with an external diameter of 150 mm, containing a hydrogen cold source. It was irradiated for 15 years in the heavy water tank of ORPHEE reactor at a mean temperature of 40°C.

In the maximal flux plane at nominal power, the mean value of the total neutron flux calculated in the cold source shell is 1.6×10^{14} n.cm⁻².s⁻¹. In the heavy water reactor tank far from the cold source, the ratio of the fast flux (E> 1 MeV) to the thermal neutron flux (E<0,625 eV) is about 100. As shown on figure 1, the presence of the cold source flattens the neutron distribution, and broadens the spectrum towards lower energies. But the total flux integrated on the energy is hardly reduced (about 4%) by the presence of the cold source.



The chemical composition of the 5754-NET alloy is 3% Mg, 0.28%Fe, 0.08%Si and traces of Zn (0.01%).

The shell is a rolled 6.3 mm thick sheet, in the annealed temper, TIG-welded over the whole length of the tube. This tube is prolonged over the core by another 12.5 mm thick tube, manufactured from a rolled sheet, and TIG-welded on the first one. After welding, all the beads were tooled to obtain a flat surface.

Tensile specimens were machined in the length of the tube, at two levels outside the weld seam: at the top of the prolongation tube used as a non-irradiated reference, and in the maximal neutron flux, just over the cold neutron source and on the side facing the reactor core. To test the same geometry at both levels, the out-flux specimens were reduced in thickness symmetrically on both sides to 6.3 mm. But the optical microscopies performed at both levels revealed a different rolling orientation: the specimens at high fluence are oriented in the rolling direction, whereas the out-flux specimens are in the transverse direction.

Micro-hardness samples were machined throughout the welded bead at both levels, in the non-irradiated state and in the maximal neutron flux. The width of the melted zone is about 2 cm in the prolongation tube, and 1.6 cm in the tube at high flux. The Vickers micro-hardness was measured at 20°C, with a 200 g weight loaded in 10 s and maintained for 10 s. On each sample, three profiles were performed through the different zones of the seam at different positions in the thickness of the tube; and one profile was made in the base metal through the thickness of the tube.

In order to estimate the thermal fluence received at each level, the silicon content was measured after abrasion of the oxide scale, by laser ablation optical emission spectroscopy on samples cut in the proof length of the tensile specimens after the tests. The non irradiated specimens from the top level contain 0.08% silicon. At the high flux level, the measured silicon content is 1.4%, which corresponds to the generation of 1.32% Si under irradiation. Considering the irradiation time of 15 years and the neutron flux of 1.6x10¹⁴ n.cm⁻².s⁻¹ calculated in a 20 cm high piece of shell centred on the cold source, this high silicon production suggests that the neutron flux in the tensile specimens could be higher or/and that the silicon generation rate is enhanced by the spectrum shift to lower energies. The cross section for neutron absorption on ²⁷Al is indeed proportional to the inverse of the neutron energy. Neutron calculations were hence necessary to evaluate the neutron flux and the silicon production rate at the position of the tested specimens.

3. NEUTRON CALCULATIONS IN THE COLD SOURCE SHELL

For these calculations [2], we used a core model validated for the Orphée reactor, by flux measurements deduced from dose integrators.

3.1. Mean values in the shell

A first calculation was performed, considering a 20 cm high piece of shell, centred on the cold source. The cold source impact on the neutron flux and on the neutron absorption rate on ²⁷Al in the shell was evaluated by comparing the values calculated in presence of the cold source inside the shell, and without cold source (replaced by vacuum). As shown in figure 1, the principal effect of the cold source is to flatten and broaden the neutron spectrum; but the total flux reduction (integrated in energy) is moderated and about 4%.

However the shift of the spectrum toward low energies has a strong impact on the neutron absorption rate on ²⁷Al; the ratio of this rate in presence of the cold source to the rate without the cold source is 1.53. As the silicon production is responsible for the major part of the damage at high Si contents, this shows that at a given fluence, the neutron spectrum received by a component can considerably modify its reactor life.

The mean neutron flux calculated in the 20 cm high piece of shell is $1,61 \times 10^{14}$ n.cm⁻².s⁻¹; but the mean calculated silicon content produced under irradiation is only 0.99% \pm 0.005%, which remains lower than the measured 1.32% content. This points out that the azimuthal location relative to the reactor core has a measurable effect on the neutron flux and on the neutron absorption rate on ²⁷Al.

3.2. Impact of the azimuthal orientation relative to the reactor core

A second calculation was hence performed by considering four independent sectors in the same shell piece centred on the cold source; the neutron flux and the neutron absorption rate were calculated separately in each shell quarter.

The neutron flux in the A sector facing the reactor core reaches 2.03×10^{14} n.cm⁻².s⁻¹, whereas it is only 1.25×10^{14} n.cm⁻².s⁻¹ in the B sector opposite to the core. The silicon formation rate reaches 2.676×10^{12} at.cm⁻³.s⁻¹ in the A sector facing the core, but 1.935×10^{12} at.cm⁻³.s⁻¹ in the B sector opposite to the core. The ratio of the neutron fluxes in both sectors (A/B) is hence 1.62, but the ratio (A/B) of the silicon formation rates is only 1.38 because the spectrum is harder in sector A.

According to this core model, after 15 years irradiation, the tensile specimens which stem from the A sector, facing the core, have received 5.07×10^{22} n.cm⁻².s⁻¹ and have produced 1.15% silicon during irradiation (which can be compared with 1.32% measured).

Even considering the azimuthal position of the specimens, the simulation still gives a lower value of silicon content produced under neutron flux, compared to the measured one. Several factors can explain this difference :

- The calculations were done on 20 cm high sectors, and the neutron flux and silicon production rate are mean values calculated on this height. The tensile specimens are indeed much shorter, and more over, the silicon content was measured punctually on samples of some mm side, cut in the middle of the proof length.
- The core model used for these calculations could be improved.

Hence a correction factor of 1.147 corresponding to the ratio "Si content measured/Si content calculated in the A sector" should be applied to the calculated silicon formation rate. We suppose that the model gives a correct evaluation of the neutron spectrum, but that the neutron flux in the proof length of the specimens is underestimated because of the 20 cm height used in the calculations.

The calculated neutron flux should therefore be corrected by a factor 1.147, to give a fluence estimation compatible with the measured Si content. On the basis of a 2.32×10^{14} n.cm⁻².s⁻¹ corrected flux, the fluence received by the tensile specimens is estimated to 5.8×10^{22} n.cm⁻².

4. RESULTS ON OSIRIS LATTICE STRUCTURE

The tensile tests were performed at 26, 100, 150 and 200°C with a static tensile device, with a strain rate of $4x10^{-4}$ s⁻¹, following the EN-NF 10002-1 standard. At each temperature, three specimens were tested.

The results reported to the unirradiated state (irradiated/unirradiated ratio) were already given and commented in [1]. The yield strength, the ultimate tensile strength, the total and uniform elongations, the necking parameter are plotted below respectively in figures 2, 3, 4, 5, 6 versus the thermal fluence (E < 0.625 eV). At room temperature, at the highest fluence, the yield strength increases by a 4.7 factor, and the UTS by a factor 2.3. The work-hardening which is high at the lowest fluence is considerably reduced at the middle fluence and reaches zero at the highest fluence where the alloy becomes brittle. The uniform elongation drops from about 18% at the lowest fluence to 3% at the middle fluence, and to values under 1% at the highest fluence.



Figure 2



Figure 3

At the lowest fluence, the temperature elevation has a favourable effect on the total elongation ; but at higher fluences, a temperature increase has a negative effect on the total elongation : at the middle fluence, it decreases gradually from 4% at 26°C, to 0.1% at 200°C ; at the highest fluence, the least temperature increase leads to a brittle behaviour.





Figure 4

OSIRIS LATTICE STRUCTURE - Tirr = 50°C - Fth(<0.625 eV)/Ffast(>1MeV) =1.2



Figure 5





Figure 6

5. RESULTS ON ORPHEE COLD SOURCE SHELL

5.1. NEUTRON SPECTRUM EFFECT

The total fluence estimated in the cold source shell specimens can be assimilated to a thermal fluence with E<0.625 eV, because the spectrum calculated in the shell (figure 1) shows that the whole broadened neutron peak is under this energy, and the fast flux is negligible ($F_{th}/F_{fast} \approx 100$).

At both fluence levels, 3 specimens were tested at 25° C, at a strain rate of $4x10^{-4}$ s⁻¹. A great dispersion was observed on the elongations measured at high fluence, which is probably in relation with the cold neutron spectrum.

The yield and tensile strengths measured on the cold source shell (red symbols on figure 7) are just slightly higher than those of the Osiris lattice structure. But the elongations are clearly lower in the cold source shell : the difference on the uniform elongation is about 4% in the non irradiated specimens between both components, and about 2% at $6x10^{22}$ n/cm² (E< 0.625 eV).

It should be pointed out that the specimens have different shapes :

- in the cold source shell, the specimen effective cross section is slightly arched (Ri = 69 mm and Re = 75.3 mm) whereas it is flat in the lattice structure, but both have the same width of 3 mm.
- The main difference is the thickness of the cross section : 6.3 mm in the cold source shell, and 3 mm in the lattice structure.





The S_0 cross section of the cold source shell tensile specimens is hence more than twice that of the lattice structure. And the proof length L_0 was chosen on each component to have $L_0/\text{sqrt}(S_0) > 5.65$. The uniform elongations are therefore comparable in both

components, and the 4% difference observable at zero fluence is not a geometry effect : it is due to the different alloy processing (forged block/rolled plate).

The elongation gap between both components decreases at high fluence, because the irradiation tends to smooth out initial microstructures. But we still have a 2% gap at $6x10^{22}$ n/cm², which are attributed to the different neutron spectrum. The data are indeed plotted versus the thermal fluence (E<0.625 eV) and all information concerning the distribution of the neutron spectrum under this energy is lost in this graphic, in particular the cold source effect. Figures 7 and 8 show that the cold neutron spectrum affects more the elongation loss than the strength increase. Moreover, this effect could be more important : the thermal fluence received by the cold source shell was estimated with the ORPHEE core model to $5.07x10^{22}$ n.cm⁻² and we applied a 1.147 correction factor to take into account the higher Si content measured. But this correction supposes that :

- the underestimation of the silicon content by the core model is due to an underestimation of the local neutron flux.
- the neutron spectrum is correctly described by the core model.

If the underestimation of the silicon content were due to an incorrect description of the neutron spectrum by the model, the thermal fluence received by the cold source shell would be lower than plotted on figures 7 and 8; hence the cold spectrum effect would be stronger.

As the prevailing damage at high fluence is caused by silicon formation during irradiation, it should be more adequate to plot the data versus the silicon content produced by neutron absorption on ²⁷Al. Figure 9 shows that for a given Si content production, the strength increase is lower in the alloy irradiated under a cold neutron spectrum. The fast neutron flux enhances the diffusion and hence has an effect on the silicon distribution and on the precipitate size.





Figure 10 shows that the elongations at high fluence are entirely correlated to the silicon content produced during irradiation, independently of the neutron spectrum: the red curves adjusted on the cold source shell results are in good agreement at high fluence with the data (black symbols) measured on the fuel lattice structure. The important parameter to evaluate the elongation loss under irradiation is therefore the silicon content produced under neutron flux, and not directly the thermal fluence.

This observation is in agreement with the results obtained on the HFR old core box [3] made of an alloy at 3.7% Mg whose chemical composition corresponds to the 5154-O alloy. Tensile tests were performed on specimens stemming from North and West walls, respectively irradiated with thermal (E<0.414 eV) to fast flux (E>0.1 MeV) ratios of 4.8 and 1.0, but having after irradiation an equal Si content of about 1.37%. The elongations measured on both walls are sensibly equal or inside the experimental dispersion : 1.5 and 1.8% for the total elongation ; 0.5 and 1.2% for the uniform elongation.

However, concerning the strength increase under neutron flux, our results on the cold source shell disagree with the common assertion "increasing the thermal to fast neutron flux ratio gives further strengthening under neutron flux". This assertion is valid only if the strength is plotted versus the thermal fluence, and it results in part from the silicon content difference. Several authors [3, 4] mention that this strengthening is beyond that expected from a simple increase in silicon level. For an equal Si content, the reference [3] relates a strong strength increase of about 66 MPa on specimens irradiated with a ratio of 4.8, compared to those irradiated with a ratio of 1.0. However, when the results are plotted versus the silicon content, the further strengthening is no more a generality. Our results on the cold source shell show indeed that a cold neutron flux can induce a slight strength decrease for a given Si content (compared to a harder spectrum with F_{th}/F_{fast} close to 1). It should be pointed out that the ratio F_{th}/F_{fast} is an insufficient information to describe the damage due to the spectrum difference :

- In the Orphée cold source shell, the fast flux (> 1 MeV) is negligible, so that there is practically no diffusion and the silicon stays where it is produced ; in the fuel lattice structure, the 0.95 ratio corresponds to a fast flux of 2.1×10^{14} n.cm⁻².s⁻¹, allowing the precipitation of the produced silicon, and leading to strengthening.
- In the HFR old core box, the fast flux on the North wall irradiated with the 4.8 ratio was about $4x10^{13}$ n.cm⁻².s⁻¹, which is sufficient to activate the diffusion ; and on the West wall irradiated with the 1.0 ratio, the fast flux was about $1.6x10^{14}$ n.cm⁻².s⁻¹. Hence the microstructures correspond to a fine dispersed precipitation on the North wall, and to a coarse precipitation on the West wall : this is in agreement with a greater strengthening on the North wall, comparative to the West wall, with equal silicon contents.

The fast flux value is therefore an important parameter, and not only the thermal to fast flux ratio : increasing the thermal/fast neutron flux ratio gives further strengthening beyond that expected from a simple increase in silicon level, only if the fast flux is sufficient to activate the precipitation.

5.2. MICROHARDNESS IN TIG-WELDING JOINS

In the base metal, the mean Vickers micro hardness measured in the thickness of the tube is 64 ± 2 HV on the out flux sample, and 168 ± 3 HV on the maximal flux sample : the 2.6 factor between these values is in agreement with the ultimate tensile strength increase under irradiation.

On the out flux sample, the micro hardness profiles through the TIG-welding join (Fig.11) present a slight decrease at the transition from the melted to the thermal affected zone : mean value of 68 HV in the melted zone against 62 HV in the thermal affected zone.



Figure 11

On the high flux sample irradiated in a cold neutron spectrum, there is still a tendency to higher microhardness values in the melted zone (about 173 HV), but the irradiation tends to smooth the transition. Moreover, the greater values dispersion at high flux masks the transition.

The mean values on the whole profiles through the welding join are very close to those in the base metal : 63 ± 3 HV on the out flux sample, and 170 ± 4 HV on the high flux sample. The ultimate tensile strength in the irradiated welding join should hence be very close to that of the base metal. So the TIG-welding is probably not affecting the loading capabilities of the base metal.

6. CONCLUSION

The Osiris fuel lattice structure irradiated at about 50°C with a conventional thermal neutron flux of 2.0×10^{14} n.cm⁻².s⁻¹ and a fast neutron flux (E>1 MeV) of 2.1×10^{14} n.cm⁻².s⁻¹ (0.95 thermal/fast ratio) becomes brittle at thermal fluences (E<0.625 eV) over 11.7×10^{22} n.cm⁻² at room temperature. At the thermal fluence of 6.2×10^{22} n.cm⁻², the alloy conserves about 4% total elongation and 3% uniform elongation at 26°C, but it decreases continuously with temperature. At 150°C, the alloy becomes practically brittle at 6.2×10^{22} n.cm⁻² with a 0.2% uniform elongation, and about 1.3% total elongation.

The Orphée cold source shell was irradiated in the heavy water at a mean temperature of 40°C in a cold neutron spectrum. In order to estimate correctly the thermal fluence and the damage in the alloy, neutron calculations were performed using a neutron core model valid for the Orphée reactor, and taking into account the perturbations of the hydrogen cold source. The broadening and flattening of the neutron peak toward cold energies leads to a higher silicon production during the irradiation. The neutron flux and the silicon formation rate are dependent on the azimuthal orientation relative to the reactor core : the silicon formation rate varies of 38% between the sector facing the core and the sector opposite to the core.

The calculated thermal fluence received by the cold source shell is rated to 5.8×10^{22} n/cm² (E<0.625 eV). A comparison of its tensile properties measured at 25°C with those of the Osiris fuel lattice structure at the same temperature shows that at this fluence :

- the strengthening is slightly increased by the cold neutron spectrum
- the elongations are about 2% lower in the alloy irradiated with the cold neutron spectrum

When considering the same data versus the silicon content produced by the irradiation, the following conclusions were drawn :

- the elongations are entirely correlated to the silicon content produced during irradiation, independently of the neutron spectrum
- with an equal Si content generated by the neutron flux, the strengthening is about 25 MPa lower in the alloy irradiated with the cold neutron spectrum
- this strengthening lack can be explained by the absence of fast flux : it is insufficient to activate the diffusion and hence the precipitation hardening.
- increasing the thermal/fast neutron flux ratio gives further strengthening beyond that expected from a simple increase in silicon level, only if the fast flux is sufficient to activate the precipitation hardening.
- the thermal to fast flux ratio is hence insufficient to describe the neutron spectrum effect : the neutron flux values are important.

Microhardness measurements through the TIG-welding join show a slight hardness increase in the melted zone. At the fluence of 5.8×10^{22} n/cm² (E<0.625 eV), the transition is smoothed by the irradiation and hardly discernible. The mean values on the whole profiles through the welding join are very close to those in the base metal : 63 ± 3 HV on the out flux sample, and 170 ± 4 HV on the high flux sample. So the TIG-welding is probably not affecting the loading capabilities of the base metal.

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