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Irradiation testing of 316L(N)-IG austenitic stainless steel for ITER

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Abstract

In the frame work of the European Fusion Technology Programme and the International Thermonuclear Experimental Reactor (ITER), ECN is investigating the irradiation behaviour of the structural materials for ITER. The main structural material for ITER is austenitic stainless steel Type 316L(N)-IG. The operating temperatures of (parts of) the components are envisaged to range between 350 and 700 K. A significant part of the dose–temperature domain of irradiation conditions relevant for ITER has already been explored, there is, however, very little data at about 600 K. Available data tend to indicate a maximum in the degradation of the mechanical properties after irradiation at this temperature, e.g. a minimum in ductility and a maximum of hardening. Therefore an irradiation program for plate material 316L(N)-IG, its Electron Beam (EB) weld and Tungsten Inert Gas (TIG) weld metal, and also including Hot Isostatically Pressed (HIP) 316L(N) powder and solid–solid joints, was set up in 1995. Irradiations have been carried out in the High Flux Reactor (HFR) in Petten at a temperature of 600 K, at dose levels from 1 to 10 dpa. The paper presents the currently available post-irradiation test results. Next to tensile and fracture toughness data on plate, EB and TIG welds, first results of powder HIP material are included.

1. Introduction

In the framework of the European Fusion Technology Programme and the International Thermonuclear Experimental Reactor (ITER), ECN is investigating the irradiation behaviour of structural materials for ITER. Austenitic stainless steel Type 316L(N)-IG, its Electron Beam (EB) welds, Tungsten Inert Gas (TIG) welds and Hot Isostatically Pressed (HIP) products are the main structural materials for the vacuum vessel and most in-vessel components for ITER. The operating temperatures of (parts of) the components are envisaged to range between 350 and 700 K. A significant part of the dose–temperature domain of ITER relevant irradiation conditions has already been explored for plate, EB and TIG welded materials, however, there is little data at about 600 K. Extrapolations of low and high temperature literature post-irradiation data tend to indicate a maximum in the degradation of the mechanical properties at this temperature, e.g. a minimum in ductility and a maximum in hardening [1–5].

The idea of application of powder and solid HIP-316L(N) in ITER is relatively new. The properties database of these materials is limited, especially when irradiation behaviour is considered. Recently ECN has performed several irradiation experiments containing powder and solid HIP austenitic stainless steel.

The experimental results of fracture toughness and tensile tests after 600 K irradiation (and a few after 350 K) at various dose levels will be presented in this paper.

2. Experimental procedures

2.1. Material and specimens

Specimens were manufactured from Type 316L(N)-IG plate material, its EB-welds, TIG-deposit and powder-HIP products. The solution annealed plate material is designated by European Reference Heats I and II

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(ERHI and ERHII). These are heats procured earlier in the European Fusion Technology program, derived from the French RCC-MR specification for the construction material of the LMFBR Super-Phenix [6]. ERHI and ERHII have minor differences in chemical composition and tensile properties [7]. An ITER Grade 316L(N)-IG has been defined, identical to the ERHI and ERHII heats [8]. Minor composition variations (impurities) are still under discussion, e.g. to reduce nuclear activation. The EB welded joints were produced from several plates of ERHII and were manufactured parallel to the rolling direction. The 316L(N) TIG weld metal was deposited according to RCC-MR practice, and is designated here as TIG-deposit [9]. The powder-HIP'ed block was produced by CEA-Grenoble from atomised 316L(N). More information on the powder-HIP procedure and techniques are presented in [10,11].

The tensile test specimens are cylindrical and have a diameter of 4 mm and a gauge length of 20 mm. For most fracture toughness tests, compact tension (CT) specimens of thickness 10 mm were used. The width of these CT-specimens is 22.5 mm, the mechanical notch is 6.5 mm. A few CT's have a thickness of 12 mm.

2.2. Irradiation experiments

The specimens were irradiated in the HFR in Petten, a mixed spectrum materials test reactor. The irradiations were performed in several series of irradiation experiments, in the so-called MANIA and SINAS type of capsules. Irradiations were performed at various dose levels (some up to 10 dpa) at approximately 600 K. MANIA experiments contain cylindrical tensile or LCF specimens, whereas SINAS capsules contain CT-specimens for fracture mechanics experiments. Both types of capsules are instrumented with thermocouples and neutron monitors, to allow calculation, by inter- or extrapolation, of the dose level and irradiation temperature for each individual specimen. Finally, a few CT-specimens of powder and solid HIP 316L(N), were included in an irradiation experiment of 2 dpa at 350 K in water (SIWAS type).

An overview of specimen types, materials, and dose level range included in this paper is presented in Table 1. Presently, a few remaining irradiations are still underway.

2.3. Post-irradiation mechanical tests

All post-irradiation tests were performed in the ECN Hot Cell Laboratory, where currently a total of 13 mechanical testing machines (including creep and miniaturised impact testing) are installed in shielded facilities. Reference tests of nonirradiated material are performed on the same machines as used for testing the irradiated specimens. All specimens were tested in air in the range of room temperature up to 700 K. Tensile tests were performed at a strain rate of 5×10^{-4}/s.

The FTT tests were performed according to the ESIS P2-91 [12] and ASTM E813 procedures. Load-line displacement is measured by means of an averaging system, whereas a direct current potential drop (DCPD) system is used for crack monitoring. The DCPD crack length measurement is calibrated by direct correlation with the final crack length as measured on both fractures surfaces (weighted nine points averaging method).

The plane-sided specimens were fatigue pre-cracked at room temperature after irradiation to avoid effects of the irradiation environment on the crack tip vicinity. Side-grooving after fatigue pre-cracking, as recommended in the ESIS/ASTM procedure, is not done for reasons of cost and handling difficulty. Moreover, the ECN practice, using the single-specimen DCPD technique without side-grooving, has shown good results: comparative experiments with side-grooved and non-side-grooved specimens of austenitic stainless steel plate material have shown that within the crack extension validity range the differences are not significant.

Finally the specimens were fractured by fatigue loading at room temperature to separate the specimen

Table 1
Overview of ECN HFR irradiation experiments

Capsule	Target conditions	Specimens	Materials
MANIA-6/7/8/9/12	600 K, 1–5 dpa, He/Ne	Tensile, Ø4 mm	316L(N)-IG plate, TIG-deposit
MANIA-10/11	550 K, 5 dpa, He/Ne	Tensile, Ø4 mm LCFØ3 mm	Solid-HIP 316L
MANIA-15	600 K, 2.5 dpa, He/Ne	Tensile Ø4 mm	Powder-HIP 316L(N)
SINAS-80/3,4,5	600 K, 1, 2, 10 dpa, sodium	CT-10 mm	316L(N)-IG plate, TIG-deposit, EB-welded
MINOSSE-1,3	600 K, 1, 10 dpa, He/Ne	CT-2.5 mm	316L(N)-IG plate, TIG-deposti, EB-welded
SIWAS-6	350 K, 2 dpa, H_2O	CT-12 mm, tensile Ø4 mm	Powder-HIP 316L(N), Solid-HIP 316L, 316L(N)-IG plate

halves without distortion of the final crack front. The initial crack length and the crack extension were measured according to the ESIS/ASTM procedure on both fracture surfaces. All crack dimension conditions were met, often except the crack front straightness condition because of nonuniform crack growth ('tunnelling'), which was severely enhanced by irradiation. The *J*-calculations were based on ESIS-P2 allowing for crack growth.

3. Results

3.1. Tensile results

The dose level dependence of the tensile properties of material irradiated and tested at 600 K is presented in Fig. 1 for plate material, for TIG-deposit in Fig. 2, and for powder-HIP'ed 316L(N) in Fig. 3. The plotted trend curves indicate the well-known irradiation hardening and reduction of ductility. For plate material at 5 dpa the 0.2% yield stress (0.2YS) practically coincides with the ultimate tensile strength (UTS), however still significant uniform (plastic) elongation is present (minimum ~5%). At 10 dpa (data taken from Ref. [3]) the UE is reduced to almost zero. The hardening is not completely saturated at 5 dpa, unlike when tested and irradiated at 500 K [7], as hardening increases another 100 to 800 MPa at 10 dpa.

The TIG-deposit behaves somewhat differently. It starts out at 0 dpa with higher 0.2YS but lower UTS (less strain hardening capacity) and less ductility than plate material [13]. The irradiation hardening builds up slower than plate, the 0.2YS of both materials is similar at 5 dpa. The strain hardening capacity is reduced to approximately zero for both materials somewhere between 4 and 5 dpa. The uniform elongation of TIG-deposit is reduced to less than 2% at 4.5 dpa. The hardening does not seem to be saturated yet.

For the powder-HIP 316L(N) specimens, all tensile properties (YS, UTS, UE and TE) except RA, are within the scatter band and just slightly below the mean curves of plate material, indicating that this material behaves much like, but not identical, to plate material. The lower data for reduction of area are also found for nonirradiated material and does not seem to degrade stronger than the RA of plate material.

3.2. Fracture toughness

An example of measured *J*-toughness curves (*J*-toughness as a function of crack-extension) is given in Fig. 4, where the *J*-toughness curves at 600 K are shown for nonirradiated and two irradiated specimens

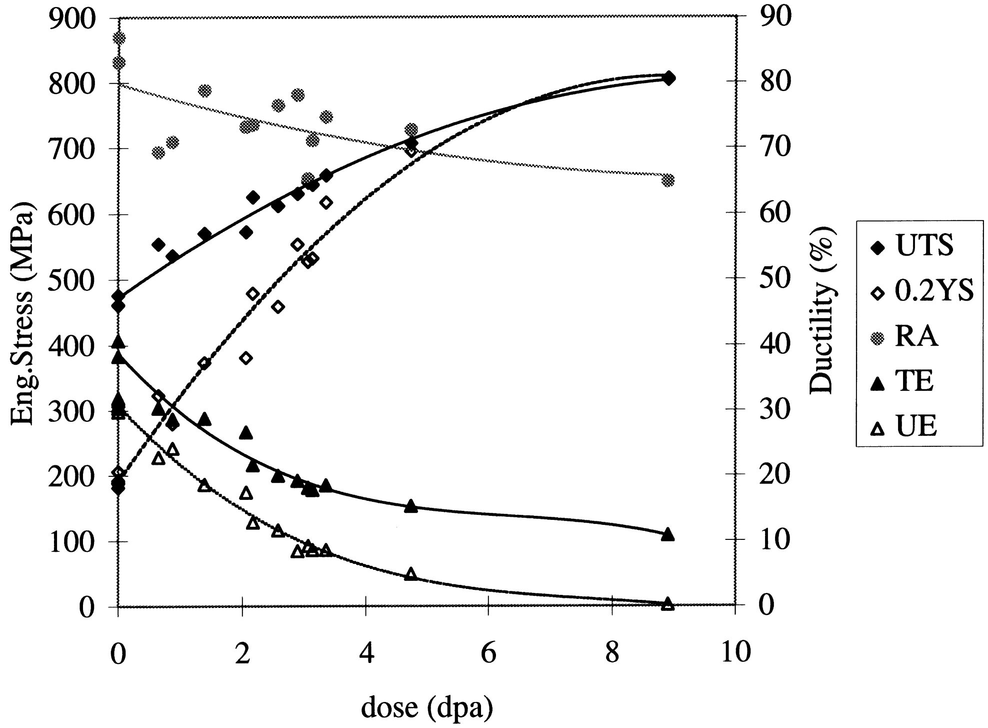

Fig. 1. Tensile properties of 316L(N)-IG plate material as a function of dose level. Irradiated and tested at 600 K.

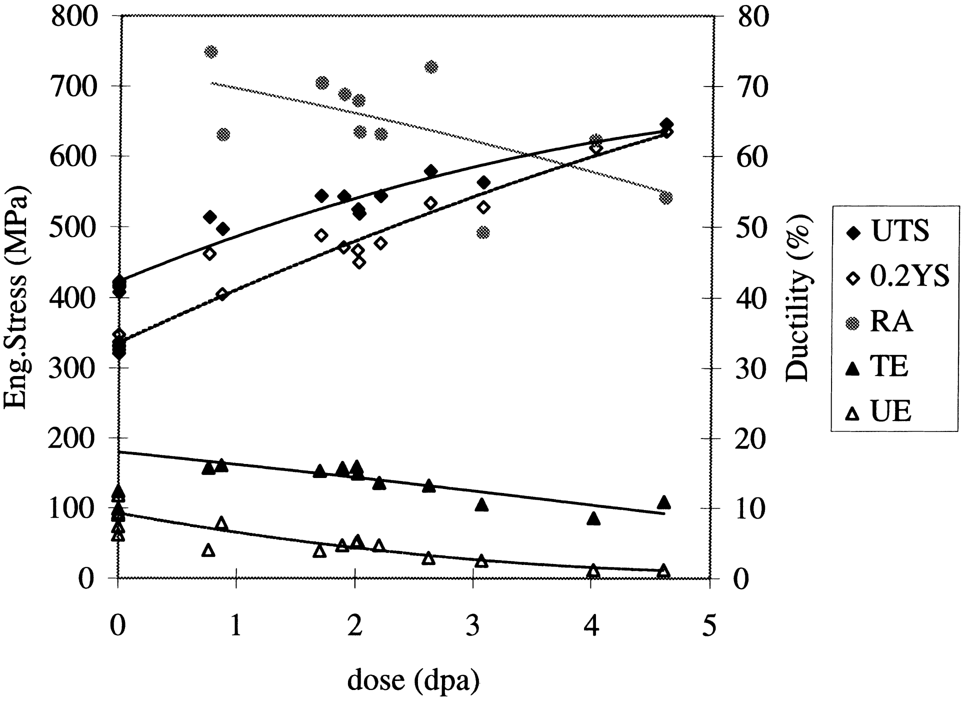

Fig. 2. Tensile properties of 316L(N)-TIG-deposit as a function of dose level. Irradiated and tested at 600 K.

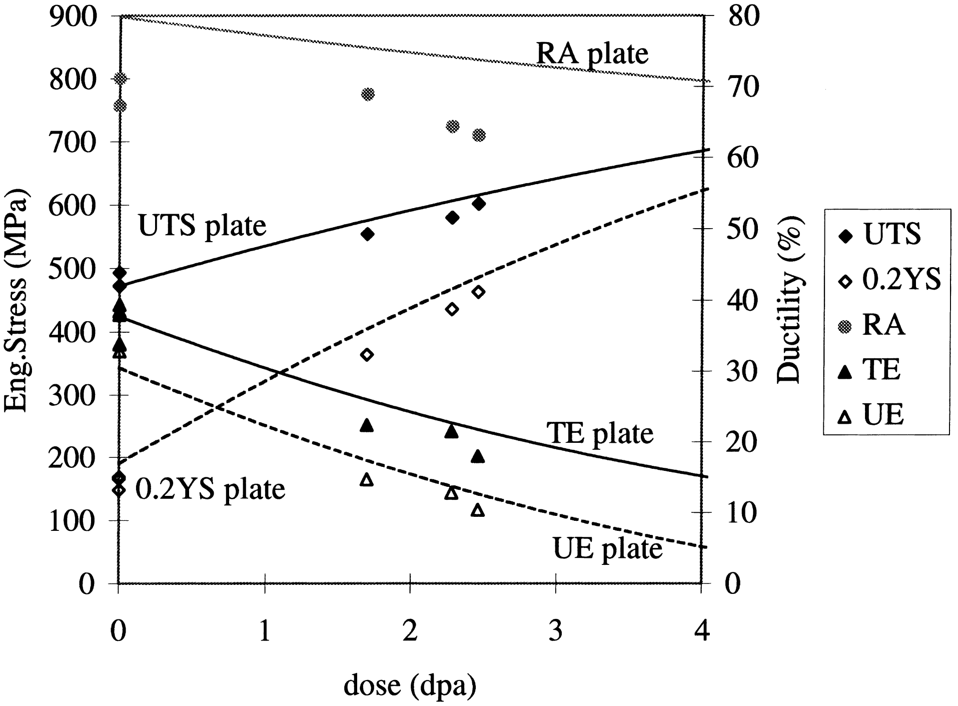

Fig. 3. Tensile properties of 316L(N) powder-HIP compared to plate material as a function of dose level. Irradiated and tested at 600 K.

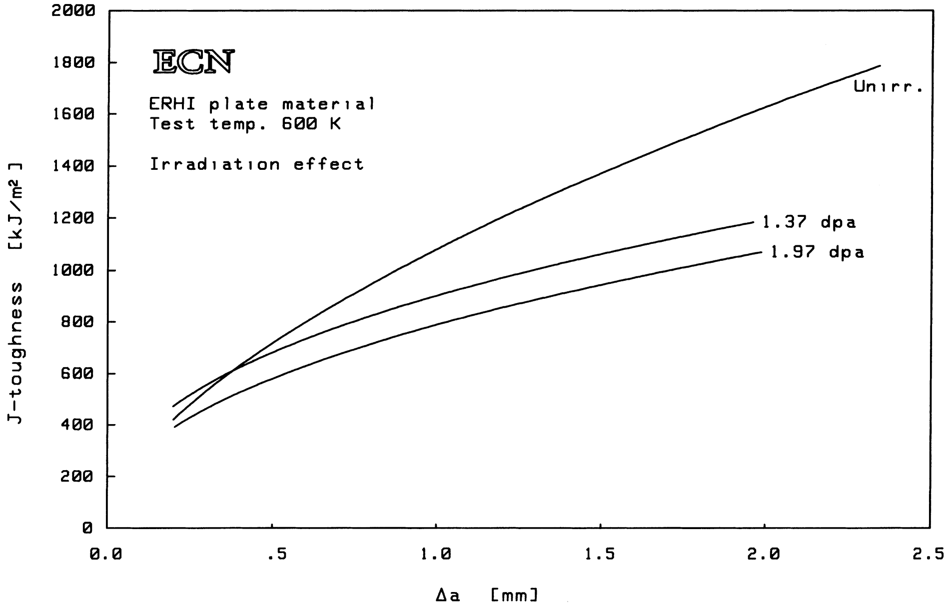

Fig. 4. *J*-toughness of 316L(N)-IG plate material as a function of crack extension and irradiation dose. Irradiated and tested at 600 K.

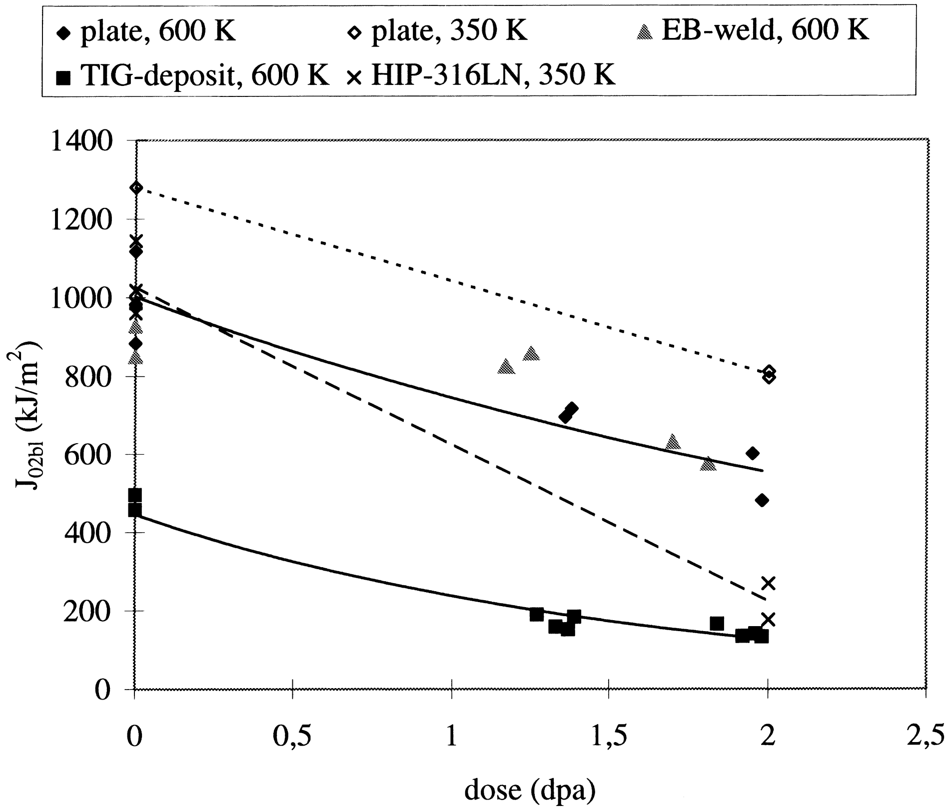

Fig. 5. J_{02BL} as a function of dose level for plate 316L(N)-IG, EB welded, TIG-deposit and Powder-HIP. Test temperature is irradiation temperature.

of plate material. Similar curves can be drawn for the other materials. From the *J*–Δ*a* curves, characteristic points can be retrieved, such as J_{02BL}. J_{02BL} is the fracture toughness at 0.2 mm crack extension, offset parallel to the blunting line. The blunting line is calculated according to the ESIS-P2 procedure, using the actual flow properties of the material. Other characteristic points can be chosen, see e.g. Ref. [14], but J_{02BL} is relatively familiar as it represents an engineering estimate of the onset of stable crack extension. Even though *J*-data can be transformed to *K*-toughness data by simple calculation, J_{02BL} is by no means similar to the usual critical elastic fracture toughness K_{IC}. K_{IC} is a critical value beyond which *unstable* fracture occurs, J_{02BL} is an estimate of the onset of *stable* crack extension. It should also be mentioned that for these very tough materials the physical crack extension (stable crack growth plus blunting) at J_{02BL} can amount up to several millimeters.

The dose level dependence of the fracture toughness J_{02BL} of plate, TIG-deposit and EB-welded material, irradiated at 600 K is given in Fig. 5. Clearly, the fracture toughness decreases with increasing radiation damage. It should be noted that the fracture toughness of nonirradiated but also low dose irradiated 316L(N)-plate is very high, usually far above the validity limits to accept the data as size independent material property.

Another observation is that the EB-welded material behaves much like the plate material, but this is only partially true. The crack propagation in the EB-welded specimens has a strong tendency to deviate out of the welded zone and continues to propagate along the fusion zones. The fracture toughness of the TIG-deposit is expectedly significantly lower than that of plate material, this is true before and after irradiation.

The fracture toughness of powder-HIP and plate material, irradiated and tested at 350 K, is also plotted in Fig. 5. The fracture toughness of unirradiated HIP'ed 316L(N) is also very high but clearly lower than that of plate material. After 2 dpa 350 K irradiation however, the fracture toughness of the powder-HIP-316L(N) has degraded stronger than plate material. It is more or less similar to that of TIG-deposit when irradiated and tested at the same temperature and at dose levels between 0.5 and 5 dpa [15]. A few power-HIP'ed 316L(N) specimens have been irradiated to 2.5 dpa at 575 K but are not yet available for testing.

The degradation of the normalized fracture toughness (normalized by dividing the toughness at 1 mm crack extension by J_{1mm} before irradiation), is plotted as a function of dose level in Fig. 6 together with various other austenitic plate and weld metal data taken from Ref. [14]. Remarkably, it turns out that the degradation of fracture toughness by irradiation is similar for many

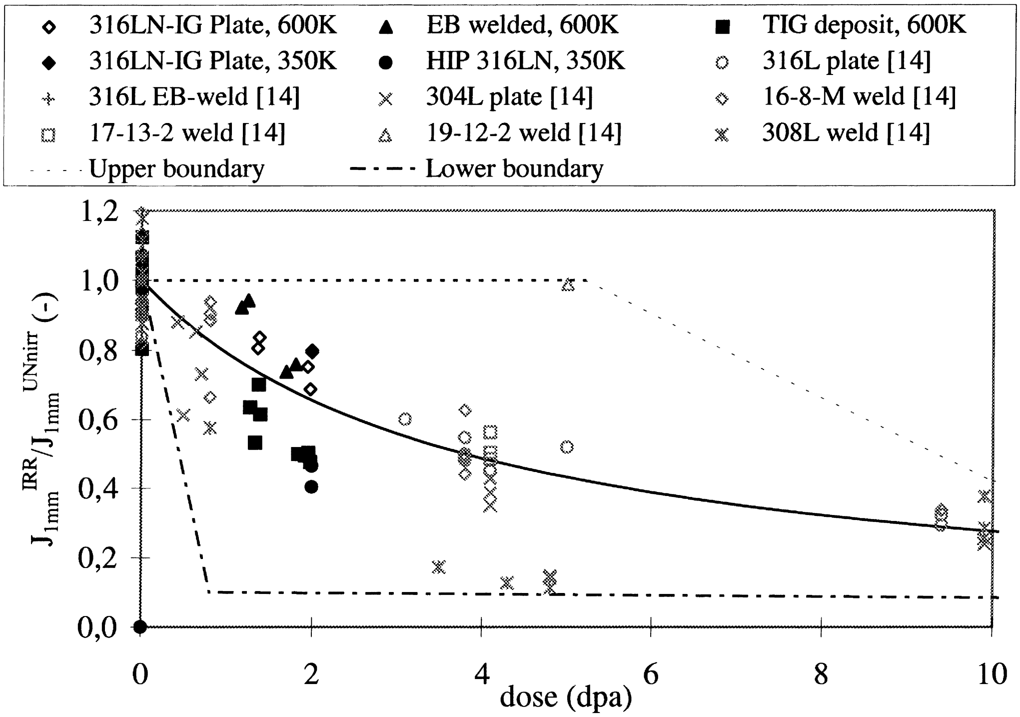

Fig. 6. Irradiation degradation of fracture toughness austenitic stainless steels and weld metals, compared to data from Ref. [14].

materials, even when irradiated and tested at very different temperatures (range 350–725K).

3.3. Microscopic observations

3.3.1. Fracture surfaces

The SEM inspection of the fracture surface of tensile test specimens revealed only ductile dimples up to 5 dpa. The powder HIP 316L(N) shows markedly smaller dimples than plate material. Preliminary results of SEM inspection of FTT-CT's show ductile dimples after 1 and 2 dpa irradiation, however the structure is already clearly different from nonirradiated.

3.3.2. TEM

Preliminary TEM-results showed black dots, developing into loops at about 2 dpa. Also small cavities have been observed after 2 dpa, these are most likely helium-bubbles.

4. Conclusions

Austenitic stainless steel Type 316L(N)-IG, its EB- and TIG-welds and its Powder and Solid HIP products are main structural materials for ITER. To fill the gap of available data after irradiation at 600 K several series of irradiation experiments have been carried out in the HFR in Petten. The mechanical properties investigated after irradiation and reported here are tensile, *J*-toughness and fatigue crack growth:

1. Strong irradiation hardening is observed in tensile tests after irradiation at 600 K. The hardening has not yet completely saturated after 5 dpa (700 MPa plate, 650 MPa TIG-deposit), but probably will do so before 10 dpa (800 MPa for plate).
2. Strain hardenability was completely lost after 4–5 dpa for plate and TIG-deposit, however uniform plastic deformation capacity (UE) remains; plate: 5%, TIG-deposit: ~2%.
3. Powder-HIP 316L(N) tensile properties after 2.0–2.5 dpa irradiation at 600 K are similar but slightly inferior to plate material.
4. The *J*-toughness of nonirradiated austenitic stainless steels is very high. The degradation due to irradiation is already severe after 2 dpa (irradiated at 600 K and at 350 K). The remaining toughness ranges from 0.5× to 0.8× the nonirradiated value.
5. The irradiation degradation of *J*-toughness of austenitic stainless steel is similar for many materials at many temperatures, as shown in a "Irradiation–Degradation Trend Curve", Fig. 6.

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