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## Austenitic Stainless Steels for Fast Reactors - Irradiation Experiments, Property Evaluation and Microstructural Studies

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### Abstract

Austenitic stainless steel SS316 and its variants are the common materials for the fast reactor structural components. Using the Fast Breeder Test Reactor (FBTR) as an irradiation test bed, a systematic analysis of the irradiation performance of the austenitic stainless steel has been undertaken. The performance of 20 % cold worked SS316 has been assessed by examining the cladding and wrapper of FBTR at various displacement damages. The modified version of SS316, alloy D9, chosen for PFBR has been subjected to test irradiations in FBTR. Further modification of alloy D9 with respect to minor elements is also being studied. The salient features of (i) mechanical and microstructural behaviour of SS316 at different fluence levels, (ii) the ongoing irradiation experiments on alloy D9 and (iii) microstructural studies on modified versions of alloy D9 are presented in this paper.

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### 1. Introduction

The economic success of Fast Reactors (FR) is dependent on the performance of the core structural material. There is a strong economic incentive to maximize the fuel burn-up in commercial reactors to reduce the fuel cycle cost enabling supply of power at a lower cost. A target burnup as high as 200 GWd/t for core structural components and 100 years of life for all permanent structurals are being attempted to achieve cost reduction. While the core components such as cladding and wrapper of the fast reactor are subjected to high fluence neutron irradiation, the permanent core structurals including components like reactor vessel, thermal shields, grid plate and other block pile components experience low fluence irradiation conditions over their lifetime.

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Major concerns in achieving high burn-up in fast reactor core components are the dimensional changes in the structural materials and deterioration of their mechanical properties associated with microstructural changes [1]. The first generation core structural material belonged to austenitic steels of type AISI 316 stainless steel. This steel has been used in FBTR up to a displacement damage of 80 dpa. Strategies to combat void swelling in austenitic stainless steel has led to the development of improved versions of austenitic stainless steels that could be used safely for exposures up to 100-120 dpa levels. Accordingly alloy D9 with appropriate modifications of Ni and Cr content with Ti additions has been developed for use as the core structural material for the upcoming Prototype Fast Breeder Reactor (PFBR). Further modification of alloy D9 (D9I) with respect to minor alloying additions namely Si and P is also being studied, in order to enhance the radiation resistance for extending the service life of core components.

Using the Fast Breeder Test Reactor (FBTR) as an irradiation test bed, a systematic analysis of the irradiation performance of the austenitic stainless steel material has been undertaken. This is done in two ways: (i) testing of material samples sourced from actual fuel clad tubes / fuel assembly wrapper tubes irradiated to various fuel burn up levels and (ii) by subjecting prefabricated specimens to desired conditions of temperature and neutron fluence levels as part of planned irradiation experiments. In addition, out-of-pile thermal aging studies are also very important for supporting and corroborating the results of experimental irradiation tests. Such studies reduce the test matrix of the irradiation experiments.

In this paper, some of the important insights obtained on the irradiation performance of SS 316 are presented. The salient features of irradiation facilities available in FBTR for irradiation of structural material specimens and development of pressurized capsules of D9 alloy for irradiation creep/swelling studies are briefly described. Finally the salient results of out-of-pile thermal aging studies on the D9I alloy are also presented.

## 2. Experience with FBTR core structural material

Type 316 Stainless Steel (316 SS) in 20% cold worked condition is used as the structural material for fuel cladding and wrapper tube in FBTR. Detailed Post Irradiation examination (PIE) has been carried out in hot-cell facilities at various fluence levels to assess the irradiation performance of SS316 structural material. The various PIE techniques employed for characterizing the irradiation damage in FBTR structural materials include dimensional measurements in hexagonal wrapper/ fuel pins, mechanical testing, void swelling measurement and transmission electron microscopic studies.

The mechanical properties of irradiated SS316 cladding were determined by remote tensile tests carried out on tube specimens sectioned from various locations along the length of the fuel pin corresponding to a combination of dpa (0-83) and irradiation temperature (430°C-500°C). The dpa variation along the length of the fuel pin was estimated from the flux and spectrum of FBTR using the NRT model [2]. The displacement damage rate of the different specimens examined is estimated to be in the range of  $0.8 \times 10^{-6}$  to  $1.6 \times 10^{-6}$  dpa/s. The tensile tests were conducted at nominal strain rate of  $5 \times 10^{-4}$ /s at temperatures corresponding to (i) reactor operation conditions (ii) fuel handling operations (180°C) and (ii) ambient conditions (25°C). It was seen that the Ultimate Tensile Strength (UTS) of the cladding showed a significant decrease at displacement damages  $> 60$  dpa both in high temperature and in room temperature tests, while the uniform elongation was around 3-4.5 % (Fig 1). Similar trends of decreasing UTS with increasing dpa have been reported by Fissolo [3] where high level of void swelling is seen to exert strongest influence on the mechanical properties due to irradiation induced precipitation. This has been analyzed to be due to the effect of high stress concentration caused by the higher density of voids leading to softening, flow localization and the associated ductility losses.

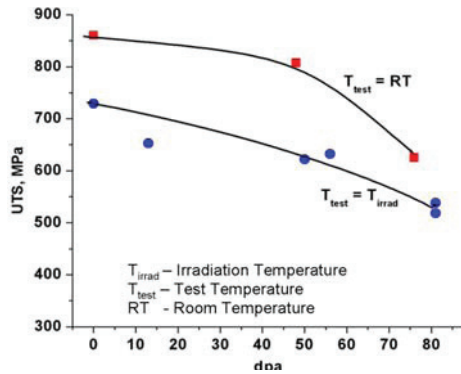


Fig 1. Trends in the UTS of cladding with dpa

The tensile properties of the hexagonal wrapper were evaluated by shear punch tests involving blanking a 1.0 mm thick and 8.0 mm diameter specimen in a test fixture using a flat cylindrical punch. Shear punch tests carried out on specimens extracted from irradiated wrapper indicated that there is an increase in the room temperature yield strength (YS) and UTS with increasing dpa (Fig. 2) and a decrease in the ductility. The tensile properties of wrapper showed a hardening behaviour as its irradiation temperature is around 400-430°C.

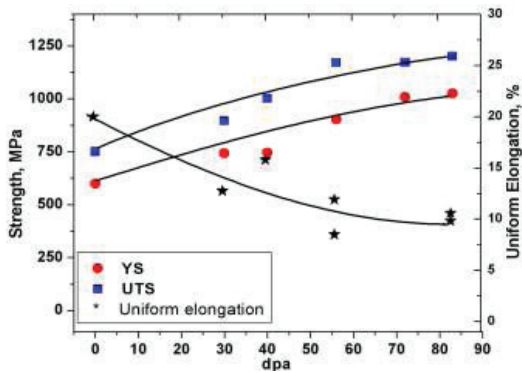


Fig.2. The trends in the strength and ductility of SS316 wrapper as a function of dpa.

Transmission Electron Microscopy (TEM) studies of hexagonal wrapper showed extensive void formation beyond 40 dpa in addition to precipitation and dislocation loops. Fig. 3 shows the TEM micrographs at different dpa superimposed on the swelling curve ( $\Delta V/V$ ) evaluated from density measurements by liquid immersion technique. The void density showed a progressive increase with displacement damage. The precipitates were identified to be mainly of nickel and silicon enriched  $M_6C$  type of  $\eta$  phase, whereas radiation induced G phase was also observed at 83 dpa [4]. The retention of cold worked structure was unambiguously seen after 83 dpa irradiation in the wrapper suggesting that no recrystallisation has taken place.

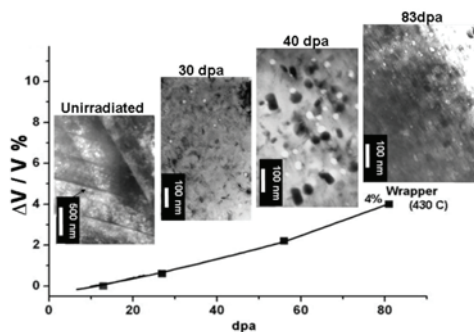


Fig 3: TEM micrographs at various dpa superimposed on swelling curve

The performance assessment of the SS 316 core structural material of FBTR by PIE was very useful in extending its use in FBTR to a burnup of 155GWd/t corresponding to a displacement damage of about 80 dpa. The main limiting factors in further extending the burnup were the void swelling of wrapper and its effect on the fuel handling operations and degradation of mechanical properties of cladding.

It can be seen that void swelling is the dominant phenomena that determine the residence time of fuel subassemblies in the core and achievable burnup. A modified version of SS316, called D9, has been chosen for core structurals of PFBR. This alloy has better swelling and irradiation creep behaviour as compared to 316 SS, which is achieved by controlled additions of silicon and titanium, increasing nickel content and lowering the chromium. The composition of alloy D9 (in wt %) is Cr:13.5-14.5%, Ni:14.5-15.5%, Mo:2%, Mn:1.65-2.35%, Si:0.5-0.75%, C:0.035-0.05% and Ti:4-6C.

### 3. Irradiation Experiments of structural materials in FBTR

Irradiation performance testing of candidate fast reactor structural materials are being carried out by subjecting prefabricated specimens to desired conditions of temperature and neutron fluence levels as part of planned irradiation experiments in FBTR. This type of experiment involves fabrication of irradiation capsules with material specimens loaded in them. Depending on the temperature required to be attained, the irradiation capsule can be of vented type (with holes in the irradiation capsule for entry and exit of reactor sodium) or gas-gap type (with gas insulation layer around the subcapsule containing specimens to attain temperatures higher than reactor sodium temperature). The irradiation capsules are locked in special subassemblies and loaded in FBTR. Normally five / six numbers of special subassemblies with irradiation capsules are loaded in the reactor simultaneously and after desired fluence levels, they are discharged from the reactor, at periodic intervals for Post-irradiation examination (PIE) in the hot cell facility. A brief description of the irradiation experiments being carried out using FBTR is given in the following sections.

### 4. Experiment with D9 alloy specimens

Pressurised capsule of D9 alloy has been developed to determine the in-reactor creep performance of indigenously developed alloy.

D9 pressurised capsules were fabricated from indigenously developed 20 % cold worked D9 alloy clad tube of outer diameter 6.6 mm and 0.45 mm wall thickness. D9 tube was closed by welding at one end and fitted with special end plug at the other end, which enabled filling of gas at the desired pressure into the tube using a pressurising system. A gas mixture of 97% argon and 3% helium was used for the pressurisation. Capsules with an internal pressure of 6.5 MPa at room temperature has been successfully developed at IGCAR. A sketch of D9 pressurised capsule is shown in Fig. 4.

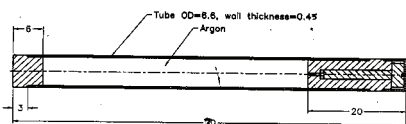


Fig.4.Sketch of D9 alloy pressurised capsule

Irradiation of D9 pressurised capsules is progressing in FBTR to determine the irradiation creep behavior at a temperature of 380°C which is the temperature of flowing sodium around the irradiation capsule. The hoop stresses that are developed in the D9 pressurised capsules at the irradiation temperature are about 30, 60 and 90 MPa respectively. The diameter changes of the pressurized capsule at these stress levels measured as a function of irradiation time, temperature and dpa will be used for evaluating the in-reactor creep of indigenously developed D9 alloy.

### 5. Irradiation of FBTR grid plate material specimens for ageing assessment

To assess the changes in mechanical properties of FBTR grid plate due to prolonged low dose exposure, an accelerated irradiation test has been carried out in FBTR on specimens of Type 316 SS. An irradiation capsule was fabricated with five compartments as shown in Fig. 5 and all the

compartments contained small size flat tensile specimens and disk specimens of type 316 SS. This irradiation capsule was assembled in a special steel subassembly with a bore of 22 mm and loaded in 4<sup>th</sup> ring of FBTR for irradiation.

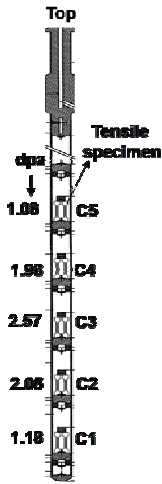


Fig.5. Irradiation capsule for grid plate specimen irradiation in FBTR

The irradiation temperature of specimens was around 350-370°C and the displacement damage ranged from 1.08 - 2.57 dpa (Fig 6). Post irradiation tensile tests were performed inside the hot cell as per the ASTM E-8 and ASTM E-21 standards at nominal strain rate of  $5 \times 10^{-4}$ /s at temperatures of 28°C, 350°C and 400°C.

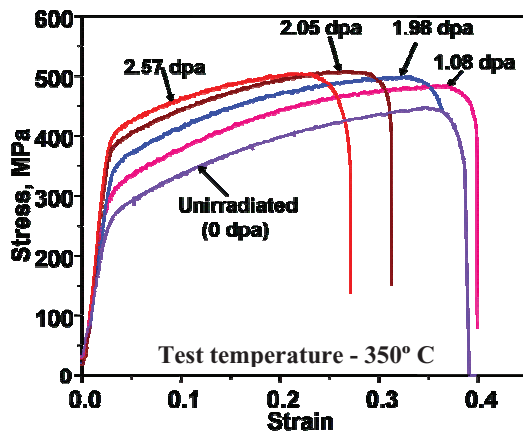


Fig.6. Stress-strain plots of the irradiated grid plate specimens of various dpa superimposed on that of virgin material.

The tensile test results of the irradiated grid plate material (SS316) show radiation hardening accompanied with ductility loss for the various dpa conditions. The narrowing of the difference between YS and UTS with increase in dpa resulted in the reduction of uniform elongation to about 20% for the specimen irradiated to 2.57 dpa. A uniform elongation of above 20% at test temperatures of 28°C, 350°C & 400°C of SS 316 indicates retention of adequate ductility in SS 316 grid plate of FBTR for an accumulated fast neutron dose of 2.57 dpa.

## 6. Development of High Temperature Gas-Gap Type Irradiation Capsule

A non-instrumented gas-gap type irradiation capsule has been developed for irradiation of structural material specimens in FBTR at temperatures higher than that of the sodium flowing around the irradiation capsule. In this type of capsule, there are five subcapsules coaxially located in the irradiation capsule and the specimens are kept in static sodium inside the subcapsules with gas gap between sub capsules and irradiation capsule. Helium-argon gas mixture with selected composition is filled in the annular gap between each subcapsule and the respective portion of irradiation capsule to attain desired higher temperatures. As part of this development, a mock up irradiation capsule containing five subcapsules has been fabricated and filled with sodium in an argon atmosphere glove box. The integrity of the sub capsules was verified by testing the subcapsules in an electrical furnace at different temperatures upto 600°C. Such gas-gap capsules are planned to be used to generate data on fast reactor core structural materials at temperatures between 400-700°C.

## 7. Studies on Modified D9 alloy

Towards developing improved versions of alloy D9 (called D9I) for clad and wrapper of PFBR, a task force has initiated studies on 20% cold worked D9 alloy with different compositions of minor alloying elements like Ti, Si and P [4]. For two different levels of P (0.025% and 0.04%) and Si (0.75% and 1.00%) at a fixed Ti:C ratio, thermal ageing studies on the four different heats of alloy (as referred in Fig. 8) were carried out at two different temperatures for a span of 1 year and 2 years period. The aim was to establish (i) the optimum alloy composition which retains the strength and (ii) the precipitate evolution that lead to depletion of the trace additions.

The hardness data for the alloys for the two different thermal ageing treatments are depicted in Fig.7. It can be seen that there is an initial increase in hardness from the as-received condition in all the alloys. This could be attributed to the precipitation of nano size MC carbides as seen in the TEM micrographs (Fig. 8).

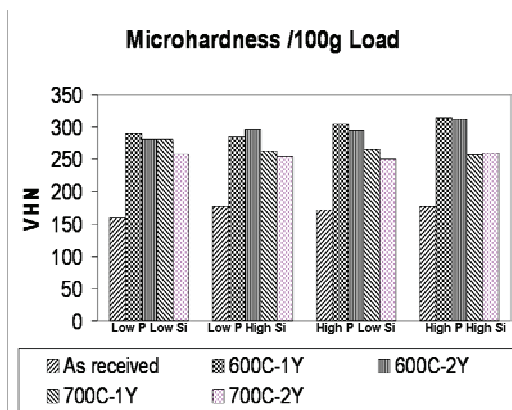


Fig .7.Hardness variation as a function of thermal ageing at 600 and 700 ° C.

The formation of carbides and intermetallics observed during thermal ageing generally depletes the elements like Cr, Mo, Ti from the matrix leading to reduction in hardness with ageing. In addition, Si is also found preferentially accommodated along with intermetallics as observed from the EDX spectrum (Fig. 9). The effectiveness of element like Si & P can be achieved only if they are retained in solution over long time of exposure at high temperatures and irradiation. Considering the need for retaining the trace elements P and Si in solution for the end-application, the low Si high P alloy is recommended. This alloy has been identified as IFAC-1.

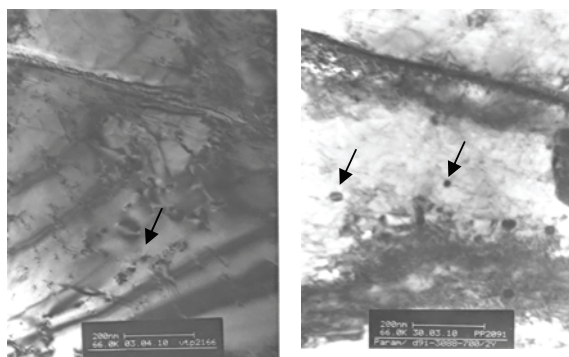


Fig 8. Evolution of TiC in high Si low P alloy: (a) 600 °C for 2 years; (b) 700 °C for 2 years

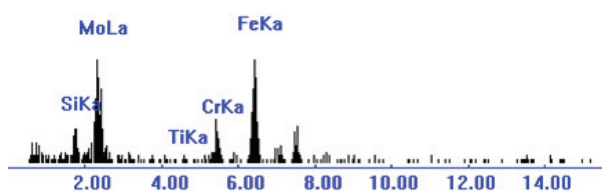


Fig 9. Energy Dispersive X ray Analysis of a precipitate of  $Fe_2Mo$  where some Si also preferentially accommodated.

## 8. Conclusions

The assessment of irradiation behaviour of austenitic stainless steel 316 in FBTR showed that void swelling and the associated dimensional changes are dominant limiting factors in extending their use beyond 80 dpa. Using FBTR as test bed, irradiation experiments on alloy D9 are under progress. Studies for development of optimized D9I are also highlighted in this paper.

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