Abstract

Development of materials plays a crucial role in the economic feasibility of fast nuclear fission and fusion power plants. In order to meet this objective, one of the methods is to extend the fuel burnup and decreasing doubling time. The burnup is largely limited by the void swelling and creep resistances of the fuel cladding and wrapping materials. India’s 500 MWe Prototype Fast Breeder Reactor (PFBR) is in advanced stage of construction. The major structural materials chosen for PFBR with MOX fuel are alloy D9 as fuel clad and wrapper material, 316LN austenitic stainless steel for reactor components and piping and modified 9Cr-1Mo steel for steam generator. In order to improve the burnup further, titanium, phosphorous and silicon contents in alloy D9 have been optimized for better swelling and creep resistances to develop modified version of alloy D9 as IFAC-1. Creep resistance of inherently void swelling resistance 9Cr-ferritic steel has been improved with the dispersion of nano-size yttria to develop oxide dispersion strengthened (ODS) steel clad tube with long-term creep strength, similar to D9, for increasing the fuel burnup. Development of modified 9Cr-1Mo steel clad tube and 9Cr-1Mo steel wrapper for future metallic fuel reactors being developed for reducing the doubling time are in progress. Extensive studies on resistance of this new generation core materials to void swelling are also under progress along with material development. Improved versions of 316LN stainless steel with nitrogen content of about 0.14 wt.% having higher creep strength to increase the life of fast reactor and modified 9Cr-1Mo steel with reduced nitrogen content and controlled addition of boron to improve type IV cracking resistance for steam generator are other developments. India’s participation in ITER programme necessitates the development of India-specific RAFM steel for Test Blanket Module (TBM). A comprehensive research programme is being carried out to develop India-specific 9Cr-W-Ta RAFM steel with the optimization of tungsten and tantalum contents for better combination of strength and toughness. Based of the extensive mechanical tests including impact, tensile, creep and fatigue on four heats of RAFM steels having tungsten in the range 1 – 2 wt. % and tantalum in the range 0.06 -.014 wt., the RAFM steel having 1.4 wt. % tungsten with 0.06 wt. % tantalum is found to possess better combination of strength and toughness. This steel is considered as India-specific RAFM steel and TBM is being manufactured by this RAFM steel. To limit the emission of green house gases, a research and development programme has been initiated to develop advanced ultra super critical fossil fuel fired thermal power plants working at temperature of around 973 K and pressure of 300 bar. High temperature creep strength super 304H austenitic steel and Inconel 617 superalloy tubes are indigenously developed for this purpose.

Keywords: Creep; low cycle fatigue; fast reactor; fusion reactor; AUSC

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1. Introduction

In India, generation of power by nuclear reactors is important because of (i) availability of a large thorium resource, (ii) constraints on setting up of fossil fuels power plants and (iii) the negligibly small greenhouse gas emissions by nuclear energy. The nuclear programme of the country is being implemented in three stages: (i) pressurised heavy water reactors of the CANDU type (ii) sodium-cooled fast reactors and (iii) thorium-based thermal and fast reactors. The second stage of sodium cooled fast reactors will provide the necessary fuel for the third stage [1]. Vast thorium reserves in the country demand the implementation of third stage. Accordingly, India has undertaken and made rapid strides in the building of fast reactors for energy generation. In India, a Fast Breeder Test Reactor (FBTR) of 40 MWt is operating successfully for over 25 years at Indira Gandhi Centre for Atomic Research, (IGCAR), Kalpakkam. Based on this experience, a 500 MWe Prototype Fast Breeder Reactor (PFBR) is under advanced stage of construction. Currently several programmes are devoted to develop new materials for higher fuel burnup with higher linear power and lower doubling time.

Sodium cooled fast reactor components are broadly classified into (i) core structural (ii) structural and (iii) steam generator. Core components comprise clad and wrapper containing fuel are critical since they are subjected to intense neutron irradiation. A comprehensive material's program has been undertaken for the development of the reactor materials. The Pu-U mixed oxide fuel will be used in this reactor. In future, Pu-U metallic fuel will be used to increase the breeding ratio to decrease doubling time.

For the successful realisation of fusion power, large international efforts are underway to develop plasma facing and breeding blanket materials which will operate at around 773 K and are capable of withstanding high neutron damage of 30 to 75 dpa/year. Reduced Activation Ferritic / Martensitic (RAFM) steels are internationally considered for the blanket material. The chemical composition of conventional grade 91 steel (9Cr-1Mo-0.06Nb-0.2V-0.05N) has been modified with the substitution of highly radioactive (induced) Mo by W and Nb by Ta to develop RAFM steel [2,3,4]. Being a partner of ITER, India has chalked out a long-term programme for the development of India-specific RAFM steel. International efforts to develop RAFM steel have focused on varying tungsten in the range 1 to 2 wt. % and tantalum in the range 0.02 to 0.18 wt. [2,3,4]. Tungsten addition increases creep rupture strength but decreases toughness properties of the steel [5]. Tantalum in the RAFM steel plays an important role in lowering DBTT through its effect on prior-austenitic grain size refinement [6]. However, higher tantalum content decreases the weldability [7]. The tungsten and tantalum contents have been optimized for better combination of strength and toughness to develop India-specific RAFM steel.

The paper presents progress and challenges in the development of fast neutron fission and fusion reactor materials being carried out at IGCAR, Kalpakkam, India. The effort towards the development of advanced ultra-supercritical material to reduce the emission of greenhouse gases is also being discussed.

2. Fast neutron fission reactor

2.1. Core structural

2.1.1. Austenitic stainless steel

Economic competitiveness of sodium cooled fast reactors (SFRs) is largely dependent on the performance of core structural materials, i.e., clad and wrapper materials of the fuel subassembly, which are subjected to intense neutron irradiation at high temperature during service. These lead to unique materials problems of void swelling, irradiation creep and helium embrittlement. Type 316 austenitic stainless in 20 % cold work condition is used for clad and wrapper in FBTR. The clad tubes in FBTR have been irradiated at the temperature range of 453 – 717 K to the fluence levels pertaining to damage levels of around 80 dpa (displacement damage). The end of life ductility was around 1 % after irradiation and extensive formation of void swelling was observed (Fig.1).

Structural materials for fast reactor core components have evolved continuously so as to improve fuel element performance. Trend in the development of radiation resistant 300 series austenitic stainless steels has been to increase nickel content and decrease chromium content in comparison to the standard versions. Solute elements like titanium, silicon, phosphorous, niobium, boron and carbon play a dominant role in determining
void swelling resistance [8]. Austenitic stainless steel alloy D9 (15Cr-15Ni-Mo-Ti-C) with specifically tailored composition, especially with regard to carbon and titanium content, has been designed around the standard AISI 316 SS to improve the void swelling resistance. This alloy in 20% cold worked condition has been chosen for the fuel clad and fuel subassembly wrapper tubes for PFBR. Creep strength of the alloy D9 is better than type 316 SS (Fig. 2). In cold worked alloy D9 SS, TiC forms preferentially on the intragranular dislocations while M₂₃C₆ precipitates on grain boundaries. TiC is more stable than M₂₃C₆ and retains its finer size over longer durations contributing to higher rupture strength and lower creep rate compared to 316 SS which is preferentially strengthened by M₂₃C₆. The fine precipitates retard recovery and recrystallisation of the cold worked structure imparting elevated temperature strength to alloy D9 [18].

![50nm](image_url)

Fig. 1. Void formation in 20% cold worked 316 austenitic stainless steel neutron irradiated at 40 dpa and 771 K.

![Comparison of creep rupture lives](image_url)

Fig. 2. Comparison of creep rupture lives of alloy D9 clad tube with 316 SS

Minor elements such as Si, Ti and P are known to have a major influence on the void swelling behaviour [8] of alloy D9. In an effort to further optimize the alloy composition around the nominal alloy D9 levels and to identify an improved version alloy D9 having higher void swelling and creep resistances, a series of laboratory heats were produced by varying the compositions of Ti, Si and P. Fifteen laboratory heats were produced with 0.025 and 0.04 w. % of phosphorous, 0.75 and 0.95 w. % of silicon and 0.16, 0.20, 0.24 and 0.30 w. % of Ti [9,10]. Influence of titanium on creep properties at 973 K showed a peak in rupture strength and a minimum in steady creep rate corresponding to Ti/C=6 in the heats containing phosphorus of 0.025 w. % and silicon of 0.75 wt % (Fig. 3). The alloys have been irradiated using 5 MeV nickel ions on (30 appm helium pre-implanted) to a peak damage of 100 dpa at a damage rate of 7 × 10⁻³ dpa/s at various irradiation temperatures between 700 and 970 K. The void swelling, measured by step height, was found to be lower for the sample containing higher amount of phosphorous and the swelling at peak temperature was 2.5% (Fig. 4). The reduction in swelling by phosphorous addition was more pronounced at temperatures > 800 K.

Based on the mechanical properties and void swelling studies on these alloys, optimized D9 alloy having Ti/C = 6 with 0.75 wt. % Si, and 0.054 wt. % P, designated as IFAC-1, is proposed for fuel pin cladding and wrapper applications. The alloy IFAC-1 with optimum composition of minor elements is expected to allow safe operation up to ~ 150 dpa for fuel clad material.
2.1.2. Ferritic steel

High chromium (9-12 Wt. %) ferritic-martensitic steels are considered as the long-term solution for fast reactor core structural materials because of their inherent void swelling resistance [11,12] and lower shift in DBTT on neutron irradiation [13]. Although these alloys [9Cr-1Mo(EM10), mod. 9Cr-1Mo (Gr. 91), 9Cr-2MoVNb (EM12), 12Cr-1MoVW (HT9) etc.] have excellent swelling resistance to doses even upto 200 dpa, (1 % swelling reported in HT9 after irradiation at 693 K at 200 dpa [11,12]), their creep resistance decreases drastically above 823 K. Therefore, they are not suitable for clad tube applications. Creep strength is not a primary requirement for the wrapper material since the operating temperatures are below the lower end of the creep range for these materials and also the stresses are low. Ferritic steels are therefore suitable for wrapper applications. However, the increase in ductile to brittle transition temperature (DBTT) due to irradiation is a cause of concern for ferritic steels. Consequently, extensive studies involving modification of the composition and initial heat treatments have been carried out to improve the fracture toughness of the ferritic-martensite 9-12 Wt. % Cr steels. Ferritic/martensitic steels containing around 9 wt. % chromium have been reported to show the lowest increase in DBTT on irradiation among the various grades of ferritic steels [13]. Effects of sulphur, phosphorous and silicon on DBTT of 9Cr-1Mo steel have been reported [14]. From low DBTT point of view, sulfur and phosphorus should be as low as possible in the ferritic steel. In an effort to specify the lower limit of silicon to lower DBTT, three heats of 9Cr-1Mo steel with silicon in the range 0.24 – 0.6 Wt. % have been melted. Variation of Cherpy-V energy with temperature of the steel is shown in Fig.5. In the investigated range (0.24 – 0.6 Wt. %), silicon has no deleterious effect on DBTT. These materials are very promising for wrapper applications and will be considered in the second stage of fast reactor along with IFAC-1 as clad material with intended burnup of more than 150 GWd/t.

To further increase the target burnup levels upto 200 GWd/t, oxide dispersion strengthened (ODS) ferritic-martensite steel Fe-0.11C-9Cr-2W-0.2Ti-0.27Y2O3 with adequate creep strength is being developed for clad tube application [15-17]. A complex powder metallurgy route followed by hot and cold mechanical processes steps was adopted to produce the clad tubes. Pre-alloyed powders of the steel and nano size Y2O3 particles were blended in a high energy simoloyer type of mill in argon atmosphere. The mixed powder was canned in mild steel can, degassed and sealed. The sealed cans were upset to compact the powder into canned billet. The upset billets were machined to remove the mild steel can. The billets were hot extruded to produce rods. Mother tubes were prepared by drilling the extruded rods with minimum concentricity. Clad tubes were produced by cold drawing (pilgering) of the mother tube in several passes with intermediate softening heat-treatments. The clad tubes were finally subjected to normalizing and tempering heat treatments in inert atmosphere. Clad-tubes with 6.6 mm outer diameter, 0.45 mm thickness and upto 4500 mm length have been successfully produced.
Figure 6 is dark field TEM image of the 9Cr-ODS steel showing Y$_2$O$_3$ particles skewed at around 10 nm. The creep rupture strength of the developed 9Cr-ODS clad tube at 973 K is shown in Fig. 7. Relatively long-term creep strength of the developed ODS steel is comparable to alloy D9. This material is very promising as clad material and will be used in the third stage fast reactor along with 9Cr-1Mo ferritic steel as wrapper material to increase the fuel burnup to around 200 GWd/t.

![Figure 6](image)

**Fig. 5.** Effect of silicon content on fracture toughness of grade 9 (9Cr-1Mo) steel.

![Figure 6](image)

**Fig. 6.** Dark field TEM image of the 9Cr-ODS steel showing Y$_2$O$_3$ particles skewed at around 10 nm.

![Figure 7](image)

**Fig. 7.** Creep rupture strength of 9Cr-ODS steel and 973 K, compared with other steels.

2.2. Structural material

2.2.1. Austenitic steel

Austenitic stainless steels of type 316 and its closely related variant 316L(N) are the preferred candidates for high temperature structural components of sodium cooled fast reactors (SFRs) due to their adequate high-temperature tensile and creep strengths, compatibility with liquid sodium coolant, ease of fabrication, weldability and commercial availability. A modified grade type 316 stainless steel has been used as the principal structural material for FBTR. This material differs from the conventional grade of type 316 stainless steel with respect tight close control of to avoid scatter in mechanical properties. Higher degree of cleanliness was achieved by specifying limits on residual elements such as S, P, B and Si and inclusion content. Despite of these measures, the alloy displayed pronounced heat-to-heat variations in the long-term creep rupture properties [18] (Fig.8). The difference in creep properties resulted from subtle variations in the amount of minor alloying...
elements (carbon 0.048-0.057 wt. %, nitrogen 0.031-0.045 wt. %, boron 0.0005-0.0015 wt. %) and grain size (0.035-0.070 microns). The improved creep resistance (of heat-A) has been associated with its finer grain size and to the relatively higher percentage of interstitials C, B, and N within the specified range. The 316SS displayed microstructural stability over long periods as depicted by linear variation in stress dependence of creep rupture life plots (Fig.9). This is attributed to the fine scale precipitation of chromium-rich \( \text{M}_2\text{C}_6 \) type of carbides on grain boundaries (Fig.9a) as well as on dislocations in the intragranular regions (Fig.9b) [19]. The fine precipitates on dislocations prevented the recovery in substructure leading to avoidance of sigmoidal relationship between stress and rupture life. While fine carbides on grain boundaries reduce grain boundary sliding, the intragranular precipitation of carbides strengthen the matrix by retarding the glide and climb of dislocations. Understanding the microstructural changes, dislocation evolution and damage mechanisms during long-term deformation in the three heats enabled the development of robust creep life prediction models that can predict lives under service conditions that are not covered by laboratory testing [20].

![Graph](image)

Fig. 8. Heat to heat variations in creep rupture strength of 316 SS at 823 K.

![Micrographs](image)

(a) (b)

Fig. 9. Precipitation of chromium-rich \( \text{M}_2\text{C}_6 \) type of carbides on (a) grain boundaries (873 K/22100 h) and (b) on dislocations in the intragranular regions (823 K/8300 h).

2.2.2. Influence of nitrogen on creep strength of 316L (N) stainless steel

In general, austenitic stainless steels have relatively poor resistance to intergranular stress-corrosion cracking (IGSCC) in chloride and caustic environments. Type 316 SS welds exposed to marine environments have been reported to fail by IGSCC in the heat-affected zone, due to the combined influence of sensitization and the presence of residual stresses introduced during welding. A nitrogen-alloyed low carbon (0.03 wt.% maximum) version of this steel (316L (N) SS) has been chosen for the high-temperature structural components of PFBR. For PFBR, nitrogen is specified in the range of 0.06 to 0.08 wt%, in order to compensate for the loss
in solid-solution strengthening due to the reduced carbon content Rupture life increased substantially with nitrogen addition (Fig.10). The beneficial effects of nitrogen arise due to higher solubility of nitrogen in the matrix than the carbon, reduction in stacking fault energy of the matrix and introduction of strong elastic distortions into the crystal lattice, giving rise to strong solid solution hardening [21]. Nitrogen also effects the diffusivity of chromium in austenitic stainless steels leading to retardation in coarsening of M_{23}C_{6} thereby retaining the beneficial effects of carbide precipitation to longer times [22, 23].

With a view to increase the design life of structural components of future sodium cooled fast spectrum reactors from 40 years to 60 years and beyond, studies are being carried out to develop a nitrogen alloyed 316LN stainless steel with superior tensile, creep and low cycle fatigue properties as compared to 316L (N) SS containing 0.07 wt.% nitrogen. The influence of nitrogen on the creep behaviour of 316LN stainless steel has been studied at nitrogen levels of 0.07, 0.11, 0.14 and 0.22 wt. % [24,25] by keeping the rest of the composition unaltered. The carbon content in these heats was 0.03 wt. %. Creep rupture strength increased substantially with increase in nitrogen content (Fig.11). After creep testing, dislocations were observed to have rearranged in the form of subgrains in the metal containing 0.07 wt. % nitrogen (Fig.12(a)). The tendency to form subgrains decreased with increasing nitrogen content. In the material containing 0.22 wt. % nitrogen, there was no evidence for the formation of cells/subgrains. Instead, the dislocations were found to be uniformly distributed in the matrix (Fig.12(b)).

![Graph showing influence of nitrogen on creep properties of 316L(N) SS steel.](image1)

![Graph showing influence of nitrogen on creep properties of 316LN SS at 923 K.](image2)

![Transmission electron micrographs of creep tested 316LN SS at 923 K. stress = 175 MPa](image3)
2.3. Steam generator materials

The niobium stabilized 2.25Cr-1Mo steel is used in the steam generator of FBTR. Modified 9Cr-1Mo steel is being used in constructing steam generators of PFBR. Moderate creep strength coupled with high thermal conductivity, low thermal expansion coefficient and virtual immunity to stress corrosion cracking in chloride and aquaduct media over those in austenitic stainless steel. Modified 9Cr-1Mo is used in the normalized and tempered condition that gives rise to tempered martensite structure. In this alloy, the additions of V, Nb and N ensure intragranular precipitation of highly stable V, Nb-carbonitrides (MX) particles on tempering and during creep exposure [26] to confer relatively high creep strength. The creep-rupture strength of indigenously developed modified 9Cr-1Mo steel in rolled, forged and tube product forms were found to be higher than the average strength values reported in RCC-MR design code (Fig.13) [27]. Steel meeting stringent requirements was produced by electro slag refining process and the forged rounds were then converted into long seamless tubes. Strict quality control was carried out at all stages including development of innovative non-destructive testing techniques.

Creep strength of the fusion welded joint of the steel is considered to be a life limiting factor. In the actual structures fabricated by welding, a high percentage of the failures have been reported to occur in the heat affected zone (HAZ) [28,29]. The detailed microstructure in the HAZ of ferritic steels is extremely complex and is controlled by the interaction of thermal fields, produced by the heat input from the welding process, and the phase transformation and grain growth characteristics of the materials being welded [30]. Further modifications in microstructure can occur as a result of tempering either during the later stages of welding and post-weld heat-treatment (PWHT) or during service. These microstructures which generally vary from wrought base material through transformed HAZs to cast weld metal, can have greatly different mechanical properties. As a consequence premature cracking occurs in the intercritical region of HAZ to reduce its creep rupture life (Fig.14), commonly termed as type IV failure. The joint of the steel possesses lower creep rupture life than the base steel (Fig.15). Chemical composition of modified 9Cr-1Mo steel has been altered with the control of nitrogen to less than 100 ppm and microalloying with boron. The steel exhibits better resistance to type IV cracking with less reduction of creep rupture strength of weld joint than the base metal (Fig.16) [31].

Fig. 13. Comparison of the creep rupture strength of modified 9Cr-1Mo steel in different product forms.

Fig. 14. Type IV failure at the outer edge of HAZ in modified 9Cr-1Mo ferritic steel weld joint (923 K, 60 MPa, T_r = 1517 hours).
3. Fusion reactor material

A comprehensive research programme is in progress to develop India-specific RAFM steel. In the first phase of the development, RAFM steel with composition conforming to Eurofer 97 was produced. Strict control has been exercised on the radioactive tramp elements (Mo, Nb, B, Cu, Ni, Al, Co, Ti) and on the elements that promote embrittlement (S, P, As, Sb, Sn, Zr, O). These elements have been restricted to ppm levels. Three heats of the steel each weighing around 200 Kg, were melted. Charpy V-notch impact properties were determined for all the three heats and for both the thicknesses of the steel using full size impact specimens. The impact specimen orientation was transverse to the rolling direction of the plate and the notch was perpendicular to the plate. The variation of impact energy with temperature showed a typical ductile-to-brittle transition curve (Fig. 17). The ductile-to-brittle transition temperature (DBTT) estimated on the basis of 68 joule criterion was less than -70 °C, as reported for Eurofer 97 [32]. Creep rupture strength of the steel is comparable to Eurofer 97 (Fig. 18).
Four heats of RAFM steel having tungsten in the range 1 – 2 wt. % and tantalum in the range 0.06 – 0.14 wt. % were melted for India specific RAFM steel. The steels were subjected to normalizing (1250 K for 30 minutes) and tempering (1033 K for 60 minutes) heat treatments and had tempered martensitic microstructure. Prior austenitic grain size was found to decrease on increase in tungsten and tantalum contents. Impact properties of the steel including ductile to brittle transition temperature (DBTT) were found to depend on tungsten and tantalum contents (Fig.19). The upper-self energy as well as DBTT of the steel increased with both tungsten and tantalum content. Tensile strength of the steel was found not to influence significantly with the increase in tungsten content, however decreased marginally with the increase in tantalum content at temperatures > 723 K with the consequent increase in ductility. Increase in tungsten content decreased the minimum creep rate and delayed the onset of tertiary stage of creep deformation in the steel and reverse was found with the increase in tantalum. Creep rupture strength of the steel was found to increase significantly with tungsten content whereas it decreased with the increase in tantalum content (Fig.20). Cyclic stress response of the steels is shown in Fig.21. Fatigue life of the steel was found to increase with the increase in tungsten and tantalum contents, however extensive cyclic softening was exhibited by the steel with a tungsten content greater than 1.4 wt. %. RAFM steel having 1.4 wt. % tungsten with 0.06 wt. % tantalum tends to have better combination of creep and fatigue strength, ductility and toughness and is considered as India-specific RAFM steel.
4. Advanced ultra super critical power plant materials

Efforts have been initiated to develop indigenously the super 304H austenitic stainless steel and alloy Inconel 617 for boiler tube applications. Tubes of both the materials have been successfully produced. Mechanical properties evaluations are currently being carried out. The developed super 304H steel has tensile and creep properties within the ± 20% scatter bands of the reported international values [33, 34]. Weld joint of the super 304H steel has been successfully fabricated both with Inconel 625 and Inconel 617 filler wire employing TIG welding process. Tensile strength of the joints was as far with the base metal value. Creep strength of the joint was lower than the base metal and failure occurred in base metal closed to the weld interface. Details of investigation are in progress.

5. Summary

Creep strength and void swelling resistance are the most important properties for fast neutron reactor core structural materials. Alloy D9 is being currently used for the clad and wrapper tubes for PFBR, under construction at Kalpakkam. To enhance the fuel burnup, composition of alloy F9 with respect to titanium, phosphorous and silicon has been optimized for void swelling and creep resistance to develop a modified alloy D9, referred as IFAC-1 SS. 9Cr-1Mo ferritic steel has been considered for wrapper application. The limit of silicon in the steel from toughness point of view has been established. The 9Cr-2W steel has been dispersed with yttria to increase its creep rupture strength at par with alloy D9 for its application as clad tube. India-specific RAFM steel has been developed with optimization of tungsten and tantalum contents for better combination of creep strength and toughness. Type IV cracking susceptibility of the weld joint of modified 9Cr-1Mo steel has been suppressed on microalloying the steel with boron and controlling the nitrogen content for steam generator applications. For advanced ultra supercritical fossil fired power plant, indigenous development of key materials such as super 304H and alloy 617 are in the process of realization.

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