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# IFE Structural Materials ARIES Assessment

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## Fusion Division Center for Energy Research

University of California, San Diego La Jolla, CA 92093-0417

## **IFE Structural Materials**

## **ARIES** Assessment

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## **Synopsis**

The HYLIFE concept utilizes a thick flibe liquid wall. The initial choice of structural material favored current steels such as 304SS which would alleviate the need for development of advanced structural materials. As part of the ARIES-IFE study, we have performed an updated assessment of the choice of the power core structural material for the HYLIFE concept. This report describes the results of this assessment.

304SS has major swelling, activation and He embrittlement issues. The swelling issue could perhaps be designed around by utilizing the wavy first wall structure assumed in HYLIFE or even with a steel fabric-type wall. The activation issue could be remedied by drastically reducing the Nb and Mo impurities in 304SS although the cost impact may be large and should be assessed. However, it is difficult to see how the He embrittlement issue could be addressed, in particular the thermal creep limits which would reduce the maximum temperature to about 550°C and thus essentially close the flibe operating temperature window for power plant application. If a 300 series SS is required as a near-term base line for the design, it is recommended that Ti-modified 316SS (PCA) be considered instead of 304SS for the first wall tubes, connecting bars and rings connecting to the back wall.! PCA has better creep and tensile strength up to about 600-650°C and is less susceptible to He embrittlement; however, it needs 2%Mo for strength which creates an activation issue even if all Nb impurity can be removed.

Moreover, it is strongly recommended that alternate structural material candidates offering the possibility of higher operating temperature and performance be considered. It should be noted that the HYLIFE concept already considers the use of unspecified advanced structural materials for some components facing more demanding conditions (such as the nozzles).

Oxide-Dispersion-Strengthened (ODS) ferritic steel and  $SiC_f/SiC$  provide the possibility of lower activation and higher temperature operation (and, thus, much better power plant performance) and are potentially attractive candidates. It is recognized that a development effort is needed in particular for  $SiC_f/SiC$  and that the data base (e.g. corrosion, erosion) must be expanded for these materials in conjunction with flibe.

#### Introduction

As part of the ARIES-IFE effort on assessing key issues and operating windows for IFE thick liquid wall concepts, the ARIES team has provided an updated assessment of the choice of power core structural material for the HYLIFE concept illustrated in Fig. 1 [1].

A number of useful discussions took place though e-mail exchanges (>80) as well as at ARIES project meetings [2,3], culminating in a recent e-meeting [4] which included several key players from the HYLIFE design study effort as well as technology and material experts (see Appendix I for details on the participants, agenda and discussion topics).

This report is based on these discussions as well as on two technical memos written by M. C. Billone as part of this assessment and included in Appendices II and III (M.C. Billone, "Evaluation of 304SS as Structural Material for IFE Thick Liquid Wall Designs," September 27, 2002; and "Evaluation of Low-Activation Ferritic Steels for IFE Thick Liquid Wall Design," October 1, 2002, Argonne National Laboratory). It aims at revisiting the reasons behind the 304SS structural material choice for HYLIFE, and at providing an updated evaluation and recommendations based on available property data to guide the selection of structural materials for such IFE concepts.



## Fig. 1 HYLIFE-II chamber concept

## **HYLIFE Structural Material Choice: 304SS**

The choice of 304SS as structural material for HYLIFE is explained in Ref. [1]. The design objective was to have a lifetime component (assumed to be 30 years) satisfying corrosion and radiation damage criteria. 300 series SS (austenitic Fe-Ni-Cr alloys) was selected for its corrosion resistance to residual TF before it gets reduced to its chemical equilibrium state of  $T_2$ . 304SS and 305SS were preferred since neither had Nb or Mo as alloying elements but only as impurities. Low activation could be obtained at the cost of removing these impurities from the steel. A dose of 100 dpa was assumed while recognizing the 300 series swelling issue. This was accommodated by making the design unusually swelling tolerant in the form of a 3-mm thick corrugated or wavy first wall which could crack or leak but not fail catastrophically. In addition and if required, the dpa level could be reduced to some extent by increasing the thickness of the flibe layer in front of the first wall and/or by increasing the size of the chamber (moving the first wall outward). The latter action can be appreciated in the context of comparing the radius of the HYLIFE-II chamber (3 m) to that of the NIF chamber (5 m). It was also believed that the choice of existing 300 series SS would avoid the extra development cost linked with newer structural materials.

The maximum operating temperature was set at 650°C to provide a reasonably high cycle efficiency. Although the 304SS strength drops rapidly with temperature, this maximum temperature was judged acceptable for the low design stress of 45 MPa. This low design stress would also enhance the fatigue life of the cyclic-stressed components.

The nozzles were a special case because of erosion concern due to the high flibe velocity of  $\sim 12$  m/s (compared to <5m/s in other parts of the power core). Based on the lifetime requirement, the nozzles could be made of special materials with relatively easily changed inserts for example or coatings. If required by erosion concern, the flibe temperature could also be decreased albeit at the cost of lower cycle efficiency and power plant performance.

## **Evaluation of 304SS as Structural Material for IFE**

## Swelling

General results from experiments on 304SS and 316SS indicate an incubation phase where swelling is negligible up to a neutron dose equivalent to  $D_o$  (incubation fluence in dpa), followed by a transient phase with a swelling rate in the range of 0-1 vol.%/dpa up to a fluence of  $D_s$ , above which a steady-state swelling rate of 1 vol.%/dpa is observed.  $D_o$ ,  $D_s$  and the transient swelling rate depend on many parameters.

Volumetric swelling ( $\Delta V/V_o$  in %) data vs. neutron dose (D in dpa) in the range of 0-10 vol.% were re-examined with an emphasis on data near a proposed design limit of 5 vol.%, on minimum values for the incubation fluence (D<sub>o</sub> in dpa) and on maximum values for the swelling rate (R in vol.%/dpa). The correlation proposed for setting an upperbound volumetric swelling for design analysis is:

 $\Delta V/V_o = R (D - D_o)$ 

(1)

With R = 0.33vol.%/dpa and D<sub>o</sub> = 10 dpa, this design correlation was compared to data based primarily on 304L SS experiments in EBR-II and was found to bound the data by at least 10-15%. In the temperature range of 390 to 530°C, D<sub>o</sub> varies from about 5 to 12 dpa and R over about 0.2-0.33 Vol.%/dpa (see Appendix II for more details).

In the range 500-600°C, the volumetric swelling is expected to be  $\leq 5\%$  at 25 dpa and roughly  $\sim 30\%$  at 100 dpa.

## He Embrittlement

Based on tests performed by Fish and Holmes (see Appendix II), irradiated (up to 35 dpa) 316SS cylindrical tensile samples show a significant reduction in total elongation as the irradiation temperature is increased from 450°C to 650°C, a recovery of ductility at 700°C and another reduction at 760-820°C (see Figure 2). The decrease in ductility from  $\approx$ 500°C to  $\approx$ 650°C is attributed to the weakening of grain boundaries due to He migration to these boundaries (i.e., helium embrittlement). As discussed by Fish and Holmes, the same effect is found in 304SS. Although not mentioned in the paper, the estimated He(appm)/dpa ratio is  $\approx$ 0.5 for both EBR-II and FFTF. Thus, He embrittlement occurs at relatively low He concentrations.

As a measure of comparison, for a HYLIFE configuration with a 85-cm thick, 42% porous flibe jet region at 50 cm from the center of the chamber and with a 460 MJ target yield and a rep rate of 4 Hz, the corresponding He production in the structure behind the jet is about 700 appm for a 40FPY lifetime. The jet region thickness would have to be increased to over 210 cm for the He concentration to fall to 1 appm (also usually considered as the reweldability limit for steel; see L. El-Guebaly's presentations, refs [3,4]).

The critical tensile property for assessing ductility and fracture toughness is the failure strain, which tends to correlate with the total elongation shown in Fig. 2. As the Fish and Holmes samples were cylindrical, the local failure strain in the neck region could be measured from the reduction in area. Reductions in area  $\geq 50\%$  suggest good fracture toughness for 304SS and 316SS. Values <10% suggest relatively brittle material and low fracture toughness. At a neutron dose of 35 dpa, the reduction in area drops to <10% for T  $\geq 650$ °C. Thermal shock resistance would be significantly reduced at  $\approx 650$ °C.

## Thermal Creep Limits

In addition to the tensile ductility decreasing to a minimum at  $\approx 650^{\circ}$ C, the creep ductility due to helium embrittlement also decreases. For these materials, which tend to have high thermal creep rates at 650°C, design criteria based on creep ductility, initiation of tertiary creep and time to rupture would limit the allowable primary stresses. For designs with very low primary stresses (e.g., fluid pressures), other limiting design criteria would come in due to secondary stresses that may cause creep ratcheting and dimensional instability. As the temperature increases, especially above 600°C, these allowable stresses are reduced significantly for long times at high temperatures due to the creep design limits. Based on creep rupture design constraints for a

design life of 10 years,  $S_m$  would be reduced from 93 MPa to 53 MPa at 600°C, and from 88 MPa to 35 MPa at 650°C for unirradiated 304SS. He embrittlement would further decrease these design allowable stresses.

If a 300 series SS is required as a near-term base line for the design, it is recommended that Timodified 316SS (PCA) be considered instead of 304SS for the first wall tubes, connecting bars and rings connecting to the back wall.! PCA has better creep and tensile strength up to 600°C, perhaps maybe even up to 650°C and is less susceptible to He embrittlement. However, it needs 2%Mo for strength which creates an activation issue even if all Nb impurity can be removed.



Fig. 2 Effect of fluence on the total elongation of type 316 stainless steel. In EBR-II, 5 dpa  $\approx 10^{22}$  n/cm<sup>2</sup>.

#### Activation

In the baseline HYLIFE-II design, the first wall is protected by 56 cm of flibe (112 cm at 50% packing fraction). The vacuum vessel (VV) wall is protected by an additional 50 cm of flibe that flows between the first wall and VV wall. The original analysis showed that most of the 304SS first wall and vacuum vessel structure would meet the waste disposal rating (WDR<1) if the Nb and Mo impurities present in "off-the-shelf" material can be removed [5]. A recent analysis has conformed that the main contributors to waste disposal rating for 304SS come from <sup>94</sup>Nb (from Nb), <sup>99</sup>Tc (from Mo), and <sup>192n</sup>Ir (from W) and that consideration of 304SS as low activation material rests heavily on the assumption of drastically reduced Nb and Mo impurity levels (< 0.005 wt.%Nb and < 0.33 wt.% Mo, as recommended by the Fast Breeder Reactor (FBR) program to reduce data scatter) (see L. El-Guebaly's presentations in Refs. [3] and [4]). Further

analyses are needed to determine the allowable impurity limits and material cost to meet those limits for a 304SS-based power core.

Note that with 56 cm of flibe, the tritium breeding ratio is 1.25 (based on 1D calculations, 1.22 based on 3D). This is higher than the ARIES power plant goal of a TBR of ~1.1 to provide enough margin for tritium breeding while avoiding excessive overbreeding. The TBR could be reduced in several ways. First, the thickness of the inner blanket could be reduced to ~50 cm (of fully dense flibe) and the flow between the first wall and vacuum vessel wall could be replaced with a steel shield to reduce breeding in that region [4,5]. Second, flibe with a reduced <sup>6</sup>Li fraction (depleted below natural Li composition) could be used (at a cost). Thirdly, Na could be added to flibe (e.g flinabe). Note that the addition of Na lowers the melting point of flibe, which has the benefit of opening the temperature operating window for the design.

## Corrosion

The desire to have high Ni content for corrosion protection is based on the fission molten salt reactor experience. However, in that case the decision was not based on compatibility with TF but with transmutation products. The natural REDOX process in that case is:

$$2UF_3 + 2TF -----> 2UF_4 + T_2$$
 (1)

Any TF in this case will be reduced. In the fusion case of flibe, it is desirable to have another chemical process to reduce TF to its elementary form. Based on the free energies of formation of TF and NiF, Ni will not be compatible with TF unless the  $T_2/TF$  ratio(redox potential) is rather high. Beryllium can reduce TF to  $T_2$  very effectively, but it is not clear whether the kinetics will be fast enough. This chemistry control process is being pursued through the JUPITER-II project.

## **Other Candidate Structural Materials for IFE**

Other possible candidate structural materials are: low activation ferritic steels, ODS ferritic steels, vanadium alloys and  $SiC_f/SiC$  composites. These are all considered part of the international MFE material R&D effort.

Low activation ferritic steels (FS) (such as the Japanese F82H alloy) have been considered in different design studies including ARIES-ST. Swelling is not an issue for FS ( $\Delta V/V_o \le 0.015$  vol.%/dpa) and helium-induced embrittlement has never been observed at high temperature although high He production (at 100 dpa) may be an issue. However, these steels lack the high temperature mechanical properties, especially above 550°C, to give the desired lifetime. The yield and ultimate tensile strengths are high at lower temperature but decrease rapidly as the temperature is increased beyond 450°C (S<sub>m</sub> = 128 MPa at 550°C, 103 MPa at 600°C, and 73 MPa at 650°C). Poor thermal creep resistance reduces these stress limits further and proves to be the life-limiting design criterion, resulting in S<sub>mt</sub> of110 MPa at 550°C and 61 MPa at 600°C for a lifetime of 3FPY (see Appendix III for more details).

Oxide-dispersion-strengthened (ODS) FS or advanced FS (AFS) provide the potential for higher temperature ( $\approx 650-700^{\circ}$ C) operation and radiation-resistance and have been developed over the last two decades as part of the Fast Breeder Reactor programs in the U.S. and Japan. The strengthening of these alloys comes from the dispersion of fine particles of Y-Ti-O. Although not optimized for fusion reactor applications, the limited database for such low-activation ODS ferritic steels suggests a shift upward of the tensile and creep properties of FS such as F82-H by at least 50-100°C (for T > 400°C).

For FS, tritium inventory (due to solubility) is increased by about a factor of 5 when compared to austenitic steel and is more of a safety concern. The ORNL assessment on the corrosion of FS with flibe suggests a maximum allowable interface temperature between FS and flibe of 700°C (see Table 1 summarizing the compatibility limits of structural materials with flibe).

Of the other MFE candidate structural materials, vanadium alloys have good strength, radiation damage and low activation characteristics and have been considered mostly in conjunction with. liquid lithium as coolant. However, they are probably not acceptable in the HYLIFE flibe case due to the large tritium inventory.

 $SiC_{f}/SiC$  composites have been considered in a number of recent MFE studies (such as ARIES-AT in the US, DREAM in Japan and TAURO in the EU) [6]. It provides attractive features such as low activation and high temperature capability for high performance power plant but needs a significant development program. There does not seem to be any feasibility issue when used in conjunction with flibe but the database is very limited and issues such as compatibility with flibe and erosion (if used in the nozzle) must be addressed by experiments.

Table 1 summarizes the different pros and cons of the structural materials for IFE use in a reactor such as HYLIFE, based on the above discussion.

	304SS	Ferritic Steels	ODS Ferritic Steels	Vanadium Alloys	SiC <sub>f</sub> /SiC
Lifetime	-	-	+	+	+
Tritium Solubility	+/-	-	-		+
Radiation Damage	-	+	+	+	+
Temperature Limits	-	-	+	+	+
Activation	+	+	+	+	+
Compatibility with Flibe	+	+	+	+	+
Material Data Base and Required Development	+	+/-	-	_	_
Considered by MFE Material Program	-	+/-	+	+	+

Table 1	Summary of	of compari	son of differen	t structural	materials for us	e in HYLIFE
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## Summary

- 304SS has major swelling, activation and He embrittlement issues. The swelling issue could perhaps be designed around such as by utilizing the wavy first wall structure assumed in HYLIFE. The activation issue could be remedied by drastically reducing the Nb and Mo impurities in 304SS although the cost impact of so doing should be assessed. However, it is difficult to see how the He embrittlement issue could be addressed, in particular the thermal creep limits which would reduce the maximum temperature to about 550°C and thus essentially close the flibe operating temperature window for power plant application. Thus, alternate structural material candidates must be considered for the power core region.
- If a 300 series SS is required as a near-term base line for the design, it is recommended that Ti-modified 316SS (PCA) be considered instead of 304SS for the first wall tubes, connecting bars and rings connecting to the back wall.! PCA has better creep and tensile strength up to 600°C, perhaps maybe even up to 650°C and is less susceptible to He embrittlement. However, it needs 2%Mo for strength which creates an activation issue even if all Nb impurity can be removed.
- ODS FS and  $\text{SiC}_{f}/\text{SiC}$  provide the possibility of higher temperature operation and much better power plant performance and are potentially attractive candidates. It is recognized that a development effort is needed in particular for  $\text{SiC}_{f}/\text{SiC}$  and that the database (e.g. corrosion, erosion) must be expanded for these materials in conjunction with flibe.
- The material development issue associated with newer materials such as ODS FS and even more  $SiC_f/SiC$  is understandable. However, even in the present HYLIFE design it is recognized that the nozzle material could be different from 304SS due to the high demands (resistance to erosion, corrosion...) placed on this component. Thus, a material development program would be needed for these anyway.
- It is important to consider the conceptual design of a fusion power plant in its proper time frame (50 years +). In this respect and in anticipation of progress by the competition and of what would be attractive to power plant operators, fusion should be looking at configurations and materials of the highest performance and safety. In MFE, only near-term experimental reactors such as ITER currently consider austenitic steel in a low fluence, low temperature environment. Austenitic steel is not considered for future reactors and is not included in the MFE materials program R&D efforts.
- For maximizing synergy between MFE and IFE R&D and to make the most of the information available from the MFE materials program, a stronger link is required between the IFE design effort and the MFE material community.

## **References**

- 1. R. Moir, et al., "HYLIFE-II: a molten-salt inertial fusion energy power plant design-final report," Fusion Technology vol 25 (1994) 5-25. See also P. A. House, "HYLIFE-II reactor chamber design refinements," Fusion Technology, 26 (1994) 1178-1195.
- 2. ARIES meeting, June 02, see http://aries.ucsd.edu/ARIES/MEETINGS/0207/
- 3. ARIES meeting, October 2002, see http://aries.ucsd.edu/ARIES/MEETINGS/0210/
- 4. ARIES e-meeting, October 2002, see <u>http://aries.ucsd.edu/ARIES/MEETINGS/0210ENG/</u>
- 5. J. D. Lee, "Waste disposal assessment of HYLIFE-II structure," Fusion Technology, 26 (1994) 74-78.
- 6. A. R. Raffray, et al., "Design and material issues for high performance SiC<sub>f</sub>/SiC-based fusion power cores" Fusion Engineering & Design," 55 (2001) 55-95

## **Appendix I**

## **ARIES E-Meeting on IFE Structural Materials**

## October 10, 2002

## List of Participants

Ryan Abbott (LLNL), <u>abbott13@llnl.gov</u> Mike Billone (ANL), <u>billone@et.anl.gov</u> Laila El-Guebaly (UW), <u>elguebaly@engr.wisc.edu</u> Wayne Meier (LLNL), <u>meier5@llnl.gov</u> Ralph Moir (LLNL), <u>Moir1@llnl.gov</u> Per Peterson (UCB), <u>peterson@nuc.berkeley.edu</u> René Raffray (UCSD), <u>raffray@fusion.ucsd.edu</u> Igor Sviatoslavsky (UW), <u>igor@neep.engr.wisc.edu</u> Dai Kai Sze (UCSD), <u>sze@fusion.ucsd.edu</u> Mark Tillack (UCSD), <u>mtillack@ucsd.edu</u>

## <u>Agenda</u>

Summary of basis for choice of 304SS as structural material for HYLIFE: (~10 minutes)	R. Moir
Summary of comparison of different structural materials: (~10-15 minutes)	M. Billone
Summary of lifetime assessment: (~5-10 minutes)	L. El-Guebaly
Discussion:	All
Wrap-up + action items:	R. Raffray

<u>Discussion Topics</u> (considering an IFE power plant that would be built in a time frame of  $\sim$ 50 years.)

What are the structural materials compatible with flibe and over what range of temperature?

304SS, FS, ODS-FS, V-alloys, SiC<sub>f</sub>/SiC....

What are the maximum temperature limits of the different structural materials (based on which limiting criterion)?

Is there a design window for operation with flibe or flinabe?

What is the lifetime for each material?

Which materials are considered by the fusion materials community?

What is the level of development required for each material?

## EVALUATION OF 304SS AS STRUCTURAL MATERIAL FOR IFE THICK LIQUID WALL DESIGNS

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September 27, 2002

#### **Summary**

Solution-annealed Type 304 austenitic stainless steel (SA 304SS) has been proposed as the structural material for IFE thick liquid wall designs. In these designs it is assumed that 304SS components are low-activation, compatible with FLiBe, and can survive the plant lifetime (30 fpy) at temperatures >600°C if the neutron dose does not exceed 100 dpa. Two radiation effects that limit the use of 304SS in a neutron environment are: stainless steel volumetric swelling and helium embrittlement. If a limit of 5 vol.% swelling is applied as a generic design criterion, then the 304SS lifetime is limited to  $\approx$ 25 dpa. The designer needs to determine how much swelling can be tolerated for the 304SS components to determine the lifetime more precisely. The upperbound design correlation recommended for 304SS swelling in the temperature range of 390-625°C and the swelling ( $\Delta V/Vo$ ) range of 0-10 vol.% is:  $\Delta V/Vo = (0.333 \text{ vol.}\%/\text{dpa}) (D - 10$ dpa) for D > 10 dpa, where D is neutron dose level in displacements per atom (dpa). At high temperatures, the lifetime of a stressed component will be limited by thermal creep strain and/or failure. However, loss of ductility due to helium embrittlement at ≈650°C and thermal creep limits set the upper temperature limit at ≈<600°C for 304SS. In austenitic steels, He mobility is high enough at  $\geq 600^{\circ}$ C to result in He concentrations at the grain boundaries. The weakened grain boundaries cause a significant decrease in creep and tensile ductility, as well as fracture toughness. The actual upper temperature limit for 304SS in an IFE design depends on the temperature and stress histories and the desired lifetime. A design lifetime of 10 years at 600°C reduces the allowable primary stress from 93 MPa to 53 MPa for unirradiated 304SS.

## 1. Introduction and Background

Fast-reactor cladding and duct material selection progressed from austenitic 304SS to 316SS to cold-worked 316SS to Ti-modified cold-worked 316SS (PCA) to ferritic-matensitic HT9 and modified 9Cr-1Mo. Solution-annealed (SA) Type 304SS (Fe-18Cr-8Ni-1.5Mn) was used very early in EBR-II as duct and cladding material for fuel rods designed for very low burnup. High swelling and creep rates limited both the lifetime and the upper temperature for 304SS components in EBR-II. Solution annealed Type 316SS (Fe-18Cr-12Ni-1.8Mn-2.5Mo) exhibited better creep resistance and appeared to be more swelling resistant based on data from low fast fluence (i.e., low neutron dose) experiments. However, as more data were accumulated at higher doses, even SA 316SS proved to be a high selling and radiation-induced-creep alloy. 20% Coldworked (CW) appeared to have optimum swelling and creep resistance for temperatures <650°C based on the performance of laboratory heats. However, in establishing tighter design

specifications on impurities (e.g., P, Si, etc.) in the cladding and duct material for the first core of FFTF, the swelling behavior of this refined heat of CW 316SS was no better than that of SA 316SS. In the case of FFTF, the assembly burnup was limited by the swelling of the hexagonal ducts made out of this Core 1 stainless steel. For higher performance in fast-reactor power designs, Ti-modified 20%CW 316SS (fusion PCA) appeared to satisfy performance requirements, along with ferritic steels such as HT9 and modified 9Cr-1Mo. Once again, even the Ti-modified steel had too high of a swelling rate for optimum core performance. The fusion heat (PCA) of this alloy exhibited high swelling with fusion-relevant He/dpa ratios. Table 1 lists the nominal compositions of the austenitic stainless steel alloys used in fast reactors and considered for use in fusion reactors. The 304SS and 316SS alloys can be grouped into categories based on the C content: "L" refers to  $\leq 0.03$  wt.% C and "H" refers to 0.04-0.10 wt.% C. 304L SS tends to be more swelling-resistant at high temperatures, while 304H SS tends to be more swelling-resistant at lower temperatures.

Garner [1] gives an excellent and thorough review of the performance of austenitic Fe-Cr-Ni stainless steels. He has shown that austenitic Fe-Ni-Cr alloys, including the 304SS and 316SS alloys listed in Table 1 approach a steady state swelling rate of  $\approx 1 \text{ vol.}\%/\text{dpa}$  in the temperature range of  $\approx 400-700^{\circ}\text{C}$ . Both the lower (300-400°C) and upper (700-800°C) transition temperatures are highly dose-rate sensitive, with lower dose rates shifting the transition temperatures to lower values. By reviewing a vast quantity of data, Garner was able to establish the sensitivity of swelling behavior to a large number of variables. Although the steady-state swelling rate does not appear to be sensitive to these variables, the incubation dose (below which the swelling is near-zero) and the dose range over which the transient swelling rate increases from near-zero to 1%/dpa are very sensitive to a wide range of parameters. For temperatures above  $\approx 475^{\circ}$ C, alloys become more swelling resistant in the incubation and transient regimes for: increasing Ni levels (20-40 wt.%), decreasing Cr levels, increasing P levels (from 0.02 to 0.08 wt.%), decreasing Ti levels (from 0 to 0.3 wt.%), decreasing C levels (0.08 to 0.02 wt.%) and increasing cold-work (0 to  $\approx 30\%$ ).

For design applications, the generic swelling limit is usually set at 5 vol.%. Specific designs may require smaller volume increases and corresponding length, thickness and width or circumference increases. Most of the austenitic alloys, including 304SS, are in the transient swelling regime for  $\leq 5$  vol.% swelling. The impact of this observation is that heat-to-heat variations in an alloy can result in significant differences in volume increase at a particular set of temperature, dpa, dpa rate, and He content. Best-estimate correlations would have to be developed for each heat of the particular steel chosen. In particular, "off-the-shelf" 304SS has composition ranges that are too broad to allow a "best-estimate" correlation to be developed with a narrow enough uncertainty band for design applications.

Element	US FBR	US FBR	US FBR	Fusion	ITER
	SA 304SS	SA 316SS	CW 316SS	CW PCA SS	SA 316L(N)
Fe	Bal.	Bal.	Bal.	Bal.	Bal.
Cr	19	17.5	17.5	14.3	17.5
Ni	9	11.8	13.5	16.6	12.3
Mn	1.5	1.5	1.8	1.8	1.8
Мо	<0.2ª	2.8	2.5	2.0	2.5
Nb	< 0.02ª		< 0.05	0.02	<0.15
Ti				0.31	
Та			< 0.02		
Si	<0.6	<0.6	< 0.75	0.5	<0.5
Cu			<0.1	0.02	<0.3
V	< 0.05	< 0.05	< 0.2	0.04	
С	0.05	0.05	0.05	0.05	0.02
Ν	0.05	0.05	< 0.01	0.008	0.07
Р	< 0.045	< 0.03	< 0.02	0.14	< 0.025
S	< 0.02	< 0.02	< 0.01	0.25	< 0.01
Со			< 0.05	0.04	< 0.25
В			< 0.003	0.001	< 0.02
Al	< 0.05	< 0.05	< 0.05	0.05	

Table 1.Nominal Compositions (in wt.%) of Types 304 and 316 Stainless Steels used in Fast<br/>Reactors and Considered for use in Fusion Reactors. SA = solution annealed and CW<br/>= 20% cold worked.

<sup>a</sup>ASME and ASTM do not generally set impurity limits on Mo and Nb for commercial applications. "Off-the-shelf" 304SS will contain Mo and Nb levels that are too high for this alloy to be classified as low activation. However, the fast breeder reactor community has set limits for Mo and Nb impurities in 304SS. For Nb, they recommend that the impurity level be <0.005 wt.% to reduce data scatter. For fusion applications in which low activation is required, tighter specifications – resulting in higher cost – need to be placed on SA 304SS.

## 2. Swelling of 304SS

Early 304SS swelling correlations [1] based on data at  $\leq 25$  dpa suggested that the peak swelling occurs at  $\approx 500^{\circ}$ C, with relatively little swelling predicted for T <  $350^{\circ}$ C and T >  $650^{\circ}$ C. However, data analyses were complicated by not properly including the early densification – as much as 0.3 vol.% for 304SS and 316SS – and the incubation fluence. These correlations generally under-predicted the steady-state swelling rate vs. T of these steels, making extrapolations to higher fluences highly uncertain.

The approach taken in this work is to re-examine volumetric swelling ( $\Delta V/Vo$  in %) data vs. neutron dose (D in dpa) in the range of 0-10 vol.%, with an emphasis on data near the proposed design limit of 5 vol.%, on minimum values for the incubation fluence (Do in dpa) and on maximum values for the swelling rate (R in vol.%/dpa). The correlation proposed for setting an upperbound volumetric swelling for design analysis is

$$\Delta V/Vo = R (D - Do)$$

(1)

Table 2 shows data and/or interpolation/extrapolation of data from Refs. 1-4 for the dpa level at which 5 vol.% swelling is reached for 304SS. For data given in the literature as a function of dose, the approximate values of Do (incubation fluence) and swelling rate (R) are also given. The data are based primarily on 304L SS experiments in EBR-II. For all points shown, the design correlation bounds the data by at least 10%.

Table 2Summary of Solution-Annealed Type 304SS Swelling Data [1-4] with Emphasis on<br/>the Dose Level Corresponding to a Volumetric Swelling of 5%

Т	Do	R	D,	Measured	Design
°C	dpa	Vol.%/dpa	dpa	$\Delta V/Vo, \%$	Correlation
					$\Delta V/Vo, \%$
390	5	0.22	28	5	6.0
434			32	5	7.3
444			36	7.5	8.7
450	8	0.22	31	5	7.0
465	12	0.33	27	5	5.7
501			34	6.7	8.0
508			27	5	5.7
530	12	0.2	37	5	9.0

A limited number of higher temperature data points were found in the literature for 304SS. Of course, much more data are available for various lots of 316SS. Table 3 summarizes the higher temperature SA 304SS data [5] as well as some of the SA 316SS and Japanese PCA (Ti modified SA 316SS) data generated as part of the fusion materials program [6]. With the exception of JPCA data at 500°C (12 dpa and 216 appm He) and 600°C (11 dpa and 517 appm He), the recommended design correlation does bound the data presented for solution annealed 304SS and 316 SS.

Table 3Comparison of Upper-Bound Design Correlation for 304SS Swelling and  $T \ge 500^{\circ}C$ Data for SA 304SS, SA 316SS and the Japanese Heat of Ti-modified, Solution<br/>Annealed PCA (JPCA)

Material	Т	D	He	Measured	Design
	°C	dpa	appm	$\Delta V/Vo, \%$	Correlation
		_			$\Delta V/Vo, \%$
SA 304SS	500	25		2.6	5.0
	600	25		0.9	5.0
SA 316SS	500	33	2057	1.1	7.6
	500	55	3445	2.3	14.5
	520	40	18	0.6	10.0
	600	36	2327	1.0	8.7
	600	41	18	0.05	10.3
SA JPCA	500	11	517	0.05	0.3
	500	12	216	2.5	0.7
	500	12	216	2.8	0.7
	500	34	2372	0.3	8.0
	500	34	2372	1.7	8.0
	500	34	2372	2.3	8.0
	500	56	3973	4.2	15.3
	500	56	3973	6.0	15.3
	500	56	3973	7.8	15.3
	520	15	6	0	1.7
	600	11	517	0.66	0.33
	600	12	216	0	0.67
	600	12	216	0.1	0.67

## 3. Helium Embrittlement of 304SS and 316SS

Fish and Holmes [7] performed tensile tests on 316SS cylindrical tensile samples that had been irradiated in EBR-II up to 35 dpa at irradiation temperatures ranging from 430-820°C. The tensile tests were performed at the estimated irradiation temperature. Figure 7 from their paper shows a significant reduction in total elongation as the irradiation temperature is increased from 450°C to 650°C, a recovery of ductility at 700°C and another reduction at 760-820°C. Figure 8 shows the mechanisms responsible for the change in ductility, as well as a plot of uniform elongation vs. temperature at 15 dpa. The decrease in ductility from  $\approx$ 500°C to  $\approx$ 650°C is attributed to the weakening of grain boundaries due to He migration to these boundaries (i.e., helium embrittlement). As discussed by Fish and Holmes, the same effect is found in 304SS. Although not mentioned in the paper, the estimated He/dpa ratio is  $\approx$ 0.5 for both EBR-II and FFTF. Thus, He embrittlement occurs at relatively low He concentrations.

From a design viewpoint, the uniform elongation has some significance with regard to design criteria. However, the critical tensile property for assessing ductility and fracture toughness is the failure strain, which tends to correlate with the total elongation shown in Fig. 7. As the Fish and Holmes samples were cylindrical, the local failure strain in the neck region could be measured from the reduction in area. Reductions in area  $\geq 50\%$  suggest good fracture toughness for 304SS and 316SS. Values <10% suggest relatively brittle material and low fracture toughness. At a neutron dose of 35 dpa, the reduction in area drops to <10% for T  $\geq 650^{\circ}$ C. Thermal shock resistance would be significantly reduced at  $\approx 650^{\circ}$ C.

In addition to the tensile ductility decreasing to a minimum at  $\approx 650^{\circ}$ C, the creep ductility due to helium embrittlement also decreases. For these materials, which tend to have high thermal creep rates at 650°C, design criteria based on creep ductility, initiation of tertiary creep and time to rupture would limit the allowable primary stresses. For designs with very low primary stresses (e.g., fluid pressures), other limiting design criteria would come in due to secondary stresses that may cause creep ratcheting and dimensional instability. Table 4 indicates the short-time allowable design stress intensities (S<sub>m</sub>) for unirradiated 304SS. As the temperature increases, especially above 600°C, these allowable stresses are reduced significantly for long times at high temperatures due to the creep design limits. Based on creep rupture design constraints Sm would be reduced from 93 MPa at 600°C to 53 MPa for a design life of 10 years.

In the absence of specific stresses and assuming that the 25-dpa limit corresponds to >7 years, it is recommended that the upper temperature limit for 304SS components in IFE designs be <600°C. A more precise temperature limit would depend on stress and design life.





Fig. 8. Effect of temperature on the uniform elongation of irradiated Type 316 stainless steel.

Table 4Short-time Allowable Design Stress Intensities (Sm) for Unirradiated SA Type 304SS<br/>based on Minimum Yield (YSmin) and Ultimate Tensile (UTSmin) Strengths. For long-<br/>time design applications, allowable stress intensities (Smt) are limited by thermal creep<br/>strain and failure criteria. Smt based on creep rupture constraints is tabulated for a<br/>design life of 10 years.

T, °C	YS <sub>min</sub> , MPa	UTS <sub>min</sub> , MPa	S <sub>m</sub> , MPa	S <sub>mt</sub> , MPa
				10 years
20	207	483	138	138
100	170	415	138	138
200	143	374	129	129
300	128	371	115	115
400	118	371	106	106
500	111	350	100	100
550	107	328	96	78
600	103	298	93	53
650	98	262	88	35
700	92	220	81	24
750	83	175	65	
800	72	132	49	

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## EVALUATION OF LOW-ACTIVATION FERRITIC STEELS FOR IFE THICK LIQUID WALL DESIGN

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#### **Summary**

Low activation (LA) ferritic steels (FS) were considered for the ARIES-ST structural material. It became readily apparent in this study that LAFS being developed by Japan (modified F82H) and the IEA (similar to F82H) lacked the high temperature mechanical properties, especially above 550°C, to give the desired lifetime of 3 years. Poor thermal creep resistance proved to be the life-limiting design criterion. As creep is a function of time at temperature, if one fixes lifetime (e.g., 3 years) and the maximum temperature (e.g., 600°C), then the thermal creep criteria will limit the stresses to 60 MPa for primary stresses due to fluid pressure and dead-weight loading, 90 MPa for combined primary-membrane and bending stresses, and 180 MPa for combined primary-membrane/bending and thermal stresses. Although only limited progress was made in the 1980's and 1990's in the development of oxidedispersion-strengthened (ODS) ferritic steels, the ARIES-ST study concluded that ODS FS showed the potential of an alloy that could be developed to meet the ARIES-ST design requirements. The properties of LAFS are summarized in this report. Progress on the development of ODS FS is also summarized. Of the two types of FS, only ODS FS has the potential for the high temperature (≈650-700°C) operation and radiation-resistance for structural material behind the thick liquid wall in the IFE design concepts.

## 1. Introduction and Background

High temperature, high-strength, low-activation, ferritic steel alloys with oxide dispersion strengthening (ODS) are considered as a relatively near-term option for the structural material of the IFE thick liquid wall designs. The structural material behind the thick liquid walls will experience a significantly reduced dose rate (dpa/year) and helium production rate (wppm/year), but it must be capable of maintaining its structural integrity up to high temperatures (650-700°C).

Considerable effort has been directed within the fusion community toward establishing design criteria for austenitic and ferritic steels [1]. However, the properties needed to apply these design criteria to ferritic steels are not included in Ref. 1. Fabrication specifications and design limits are contained in both the U.S. ASME Boiler & Pressure Vessel Code [2] and the French RCC-MR [3] for modified 9Cr-1Mo (Fe-9Cr-1MoVNb). As the RCC-MR Code has developed design guidelines for these steels up to higher temperature (600°C) than the ASME Code, it is used here as a reference. The major alloying constituents of the code-qualified ferritic steel are: Fe-(8-9)wt.%Cr-(0.85-1.05)wt.%Mo-(0.18-0.25)wt.%V-(0.06-0.10)wt.%Nb. Table 1

shows the RCC-MR minimum ultimate tensile strength (UTS), minimum yield strength (YS) and design stress intensity factor (S<sub>m</sub>) for this ferritic steel, along with the creep-limited design stress intensity (S<sub>mt</sub>) for a three-year design life. The tensile and thermal creep data for the Japanese F-82H and the IEA heat F-82H (mod) were compared to the data for the RCC-MR code-qualified modified 9Cr-1Mo. Even with the irradiation-induced softening of ultimate tensile and yield strengths of the F82-H above ≈400°C, the tensile properties of F-82H remained within the scatter band of those for Fe-9Cr-1MoVNb. Also, the minimum thermal creep strength properties of modified 9Cr-1Mo provide a reasonable lower bound for those of F-82H and F-82H(mod). Thus, the established design-limit stresses and criteria for the code-qualified modified 9Cr-1Mo can be used as a reasonable lower bound for the new low activation steels. [4] The short-term tensile properties result in a design stress intensity limit of 148 MPa at 500°C, 128 MPa at 550°C and 103 MPa at 600°C. However, thermal creep for 3 full-power-years of operation limit these stresses to 110 MPa at 550°C and 61 MPa at 600°C. The design stress intensity directly limits the average (through-wall) primary stress and indirectly (though multiplicative factors) limits the primary bending and secondary thermal stresses. A more complete description of the design limits of low-activation ferritic steels is given by Billone in Ref. 5.

Because of the low thermal creep strength of traditional and low-activation ferritic steels in the temperature range of 550-600°C, the ARIES-ST design team selected an oxide-dispersionstrengthened (ODS) ferritic steel. These alloys were developed as part of the Fast Breeder Reactor programs in the U.S. and Japan. The strengthening of these alloys comes from the dispersion of fine particles of Y-Ti-O. Although not optimized for fusion reactor applications, the compositions (in wt.%) of the low-activation ODS ferritic steel alloys tested in the 1990's were in the range of Fe-(11-14)Cr-(2-3)W-(0.4-0.5)Ti-(0.2-0.7)Y<sub>2</sub>O<sub>3</sub>, with some extra oxygen ( $\approx$ 0.1 wt.%) added. Given the limited database for such low-activation ODS ferritic steels, the tensile and creep properties of F82-H were shifted (upward) by 50°C (for T > 400°C) for design analysis. Data reported by of Mukhopadhyay et al. [6] suggest that such a shift is reasonably conservative, from a design perspective, for the thermal creep properties of ODS-FS. The assumed UTS, YS, S<sub>m</sub> and thermal-creep-limited (three-year lifetime) S<sub>mt</sub> for ODS ferritic steel can be derived from Table 1 with the simple shift of 50°C for ODS FS.

The simultaneous effects of neutron damage and He transmutation on the DBTT of FS are not well established at T< 400°C. However, the embrittlement observed in FS below 400°C are not relevant to IFE thick liquid wall design concepts. The relevant radiation effects for FS above 400°C are swelling and irradiation creep. Garner et al. [7] show that ferritic steels such as HT9 and modified Fe-9Cr-1MoVNb have a stress-free swelling rate of 0.015%/dpa up to 200 dpa in the temperature range of 400-425. The irradiation creep rate was found to be proportional to stress with a coefficient of  $2.19 \times 10^{-4} %/(dpa MPa)$ . Neither of these effects would limit the use of ferritic steels in the IFE thick liquid wall conceptual designs. Table 1 Comparison of Minimum Yield  $(YS_{min})$  and Ultimate Tensile  $(UTS_{min})$  Strength, Short-Term Allowable Stress Intensity  $(S_m)$  and Time-Dependent Stress Intensity  $(S_{mt})$  for Low-Activation Ferritic Steel (e.g., Modified F82H or IEA Heat). Note: primary stresses due to gas pressure and dead weight loading are limited by  $P_m \le S_{mt}$ , primary bending stresses are limited by  $P_m + Pb \le 1.5 S_{mt}$ , and secondary thermal and bending stresses (Q) are limited by  $P_m + P_b + Q \le 3 S_{mt}$ 

Т	YS <sub>min</sub>	UTS <sub>min</sub>	S <sub>m</sub>	$S_{mt}(3 \text{ years})$	$S_{mt}(30 \text{ years})$
°C	MPa	MPa	MPa	MPa	MPa
400	338	474	174	174	
450	320	441	162	162	
500	293	483	148	148	
550	255	417	128	110	
600	203	339	103	61	
650	137	196	73	31	

#### 2. Assessment of Oxide-Dispersion-Strengthened (ODS), Low-Activation Ferritic Steels

Commercial oxide-dispersion strengthened (ODS) ferritic alloys MA956 and MA957 were evaluated for use as fast breeder reactor (FBR) cladding [8]. Table 2 summarizes the composition of M957, along with developmental alloys currently under consideration for use as fusion reactor structural materials. The Mo content ( $\approx 0.3 \text{ wt.}\%$ ) of M957 may be too high based on activation considerations. Molybdenum, along with titanium, is added as an alloying element to improve strength, ductility and oxidation resistance. The oxide dispersion is accomplished by adding very fine particles of Y<sub>2</sub>O<sub>3</sub> to the base composition. The alloy is highly anisotropic and the properties are very sensitive to heat treatment. The Ti appears to be mostly in the form of TiC, which affects the grain structure. The anisotropy is associated with sub grains that are about 0.5 µm wide of 5 µm long (extrusion direction). In general, tensile strength and creep strength of M957 is better in the axial (drawing) direction than in the transverse (hoop direction for tubes) direction.

Table 2 lists one (LAF-3) of many alloy compositions being studied at ORNL [9]. The base composition is similar to that of F-82H. The amount of  $Y_2O_3$  and  $TiO_2$  added to the base composition is a variable with the total amount varying between 0.25 to 1.00 wt.% and the molar ration of  $TiO_2/Y_2O_3$  varying from 0 to 2. The composition of the alloy (LAF-3) with the highest tensile strengths vs. temperature is listed in Table 2.

ODS FS alloys have been fabricated by Kobe Steel Ltd. in Japan and are being tested at ORNL [10]. Two base compositions (12Y1 and 12YWT) are used, with variations in Ti, Y and O. Both alloys contain about 0.25 wt.%  $Y_2O_3$ . However, 12YWT (shown in Table 2) appears to have superior tensile and creep strength at high temperature (<900°C). Also shown in Table 2 is the composition of the ODS FS being developed by PNC for fission and fusion applications. [11]

Table 2Chemical Compositions of Commercial (M957) and Developmental Oxide-Dispersion-<br/>Strengthened (ODS) Ferritic Steels. Impurity levels in parentheses are based on<br/>maximum levels established for IAE heat of modified F-82H. NS = Not Specified.<br/>The ODS FS based on EUROFER is a hypothetical composition.

Element	MA957	Based onBased onIEA HeatEUROFERLAF-3		Japanese 12YWT	PNC 1DS
Fe	Balance	Balance	Balance	Balance	Balance
Cr	13.5-14.2	8.6	8.8	12.6	11.0
Ti	0.95-1.38	0.18	0.05-0.18	0.35	0.40
Y	0.19-0.28	0.6	0.1-0.6	0.16	0.51
0	0.006-0.240	0.26	0.03-0.26	0.16	0.21
V	NS	0.29	0.19	NS	NS
С	0.012-0.017	0.065	0.10	0.052	0.09
Mn	0.05-0.12	0.44	0.37	0.05	0.03
Та	NS	0.08	0.068	NS	NS
W	NS	2.0	1.1	2.44	2.67
Si	0.02-0.07	0.24	0.005	0.1	0.05
N	0.025-0.080	(<0.02)	0.021	NS	0.01
Ni	0.10-0.15	(<0.1)	<0.005	0.27	0.15
Мо	0.28-0.32	(<0.05*)	< 0.005	NS	NS
Nb	NS	(<0.0002)	< 0.001	NS	NS
Р	0.004-0.030	(<0.01)	< 0.005	NS	0.003
Sn	0.002		< 0.02	NS	NS
S	0.004-0.006	(<0.01)	0.003	NS	0.002
Cu		(<0.05)	< 0.005	NS	NS
Al	0.055-0.17	(<0.01)	<0.01	NS	NS
As	< 0.0001	NS	< 0.02	NS	NS
B	< 0.005	(<0.001)	< 0.001	NS	NS
Co	NS	(<0.01)	< 0.005	NS	NS

In assessing the applicability of low-activation ODS FS alloys for IFE structural material, the important points to consider are: cost of fabricating special shapes such as nozzles, optimization of heat-treatment and drawing process (called Thermo-Mechanical Treatment –TMT) to produce an isotropic alloy, strength vs. ductility, and radiation performance. In general, the higher the tensile strength, the lower the ductility, and the more brittle the alloy. For components that do not call for very high strength, high ductility and fracture toughness are desirable from both a fabricability perspective and a performance perspective. The average tensile properties [yield strength (YS), ultimate tensile strength (UTS) and total elongation (TE)] are compared at high temperature for F82H and two ODS ferritic steels at T  $\geq$  500°C. Also shown in the table is the short-time design stress intensity (S<sub>m</sub>, allowable primary stress) for these alloys. As S<sub>m</sub> is defined in terms of minimum tensile properties and ferritic steels exhibit much larger heat-to-heat variation in tensile properties than austenitic stainless steels, the RCC-MR definitions relating minimum-to-average values are used here: S<sub>yd</sub> = 0.73 YS<sub>avg</sub> and S<sub>ud</sub> = 0.83 UTS<sub>avg</sub>. For ferritic steels used as pressure vessel and piping components, S<sub>m</sub> = Min {(2/3) S<sub>vd</sub>, (1/3) S<sub>ud</sub>}.

Both ODS ferritic steels have better strength properties than F-82H, especially at higher temperatures. The alloy 12YWT is stronger than LAF-3. Based on the initial set of thermal creep data, the creep resistance of 12YWT is better than that of F-82H and the commercial ODS alloys M956 and M957. Remaining challenges in ODS alloy development are optimizing the TMT needed to maintain near-equiaxed grains and, hence mechanical property isotropy, demonstrating stability and ductility in a fusion-relevant neutron environment, and product forming and joining.

Table 3Comparison of Average Tensile Properties of Low-Activation (LA) Ferritic Steel (FS)<br/>Alloys without (F82H) and with (LAF-3, 12YWT) Oxide Dispersion Strengthening<br/>(ODS). YS = average tensile yield strength, UTS = average ultimate tensile strength,<br/>TE = Total (Plastic) Elongation, and  $S_m$  = Design Stress Intensity Limit for Primary<br/>Membrane Stresses in Pressure Boundary Components.

Alloy	Т	YS	UTS	TE	RA	Sm
5	°C	MPA	MPA	%	%	MPa
F-82H	500	439	482	18	85	133
	550	366	427	19	89	118
	600	300	366	24	93	101
	650	220	300	27	95	83
	700	134	232	30	95	64
ODS						
LAF-3 [9]	500	730	970	13		268
	650	380	480	22		133
	700	290	400	17		111
ODS						
12YWT	500	1030		8		≤500
[10]						
	550	950		8		≤460
	600	870		9		≤420
	650	450		12		≤220
	700	430		20		≤210
	750	350		17		≤170
	800	320		11		≤155
	900	210		10		≤100

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