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Progress and critical issues of reduced activation ferritic/martensitic steel development

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Abstract

The inherent properties of reduced activation ferritic/martensitic (RAFM) steels include reduced swelling and high recycling potential, which make them likely candidates for application in commercial fusion power plants. The International Energy Agency (IEA) agreement has been an effective framework for international co-operation in developing RAFM steels. The progress and critical issues observed in this co-operation are reported. The production of RAFM steels on an industrial scale has been demonstrated. Various methods of fusion welding and solid hot isostatic pressing (HIP) are feasible for joining the steels. Manufacturing of complex shapes with the powder HIP method works well for RAFM steels. Major critical issues addressed concern the effects of simultaneous introduction of helium and displacement damage. The availability of a 14 MeV neutron source is identified as an essential tool to determine this effect. Finally, the potential of oxide dispersion strengthening to increase the operating temperature of RAFM steels. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Energy from the fusion of atomic nuclei has the potential to contribute a major fraction of the world's growing power needs. A recent forecast concluded that in the second half of the next century fusion power plants might generate electricity commercially to provide a significant amount of the world's power requirements [1]. Whether the basic principle is magnetic or inertial confinement is not yet quite clear. What is sure, is that materials operating near the fusion reactions will be exposed to a high flux of high energy neutrons.

From the beginning of the attempts to harness fusion power, it was evident that the availability of appropriate materials to operate near the power source is crucial. Early work was aimed at the evolution of the steels that performed well in fission reactors. Examples are the austenitic steels, the more conventional and the low activation variant. Limitations from swelling, inferior thermal properties, helium embrittlement and microstructural instabilities forced investigators to explore other paths. The ferritic/martensitic steels do not suffer (or suffer less) from these phenomena than the austenitic steels, and they can be made in reduced activation compositions. During the last decade, the development of reduced activation ferritic/martensitic (RAFM) steels has been actively pursued [2]. RAFM steel is presently the most realistic contender for application in fusion blankets near a magnetically confined plasma. SiC/SiC ceramic composites and vanadium and chromium alloys

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could become competing candidates, because of their high-temperature properties. The development of these materials has to advance considerably before they have reached the stage where RAFM steels are today. The track record of conventional ferritic/martensitic steels in fission reactor cores also supports the RAFM development direction [3].

The framework of the International Energy Agency (IEA) implementing agreement with the partners from Japan, the US and the EU proved beneficial in the development of the steels. In the larger part of the last decade, the progress reaches from laboratory-scale castings to industrial-scale reduced activation steel production. The IEA workshops held to evaluate the progress and to discuss the plans proved to be most instrumental in that progress. The co-operation speeded up the pace and utilized the material and limited technical/scientific resources most effectively. In addition, the ICFRM symposia have promoted the exchange of facts and ideas. This ICFRM-9 conference includes about 40 contributions devoted to RAFM steel developments.

The present paper reviews the major progress and critical issues of the RAFM development since ICFRM-8. It addresses the research results and the contributions of the industry to steel production and manufacturing capabilities with this class of steels. Characterization of the steel microstructure and the effect of neutrons on its properties are major items. Further the future of the RAFM steels, development is outlined. The potential use of oxide dispersion-strengthened (ODS) steels to increase the upper temperature limit for application is also discussed.

2. Design requirements

The successful operation of the blanket structure near the plasma determines to a large extent the commercial success of a fusion power plant. The plant reliability, refuelling down time and waste production depend on the major substances in the blankets. The maximum allowable temperature in the blanket determines the thermal efficiency of the plant. Therefore, the structural material in the blanket forms a major constituent in the successful outcome of the choices to be made.

Present concepts, such as the helium-cooled pebble bed (HCPB) and the water-cooled lithium-lead (WCLL) blankets, to be tested integrally in ITER or its follower, have been designed with RAFM steel [4]. These blankets reach technical specifications that are acceptable today, but in view of the competition it is preferable to have room for an increase of the operating temperature. The oxide strengthening of RAFM steel might provide the extra push. This does not necessitate the construction of the whole blanket from this advanced steel. Only in the high-temperature regions of the blankets would its properties be needed. Although impurity levels in steel can determine the reduced activation characteristics of the RAFM steels, the impurities need not be similar over the whole blanket as the neutron spectrum rapidly changes with distance from the plasma.

These design considerations form a driving force for pushing the application temperature upwards. They also result in a quest for a palette of manufacturing, repair and replacement techniques to assure component reliability and fast refueling. Under the IEA agreement therefore, several lines of research and development (R&D) have been instituted to address these requirements.

3. Production

3.1. Steel half products

In the EU an industrial-scale heat of EUROFER97, an RAFM steel, has been delivered. The chemical analyses have not been completed, but preliminary results are given in Table 1. The batch includes plates of several thicknesses and rods partly for atomization processing of powder. Wires for TIG welding are also included.

Table 1

Chemical composition (in wt%) of EUROFER97 (preliminary data)^a

Element	Measured	Targets		Remarks
		Min	Max	
Cr	8.82	8.50	9.50	
С	0.10	0.09	0.12	
Mn	0.37	0.20	0.60	
Р	< 0.005		0.005	
S	0.003		0.005	
V	0.19	0.15	0.25	
N_2	0.021	0.015	0.045	
O_2	0.0026		0.01	
W	1.1	1.00	1.20	
Та	0.068	0.05	0.09	
Ti	0.006		0.01	
Si	0.005		0.005	
Nb	< 0.001			ALAP<0.001
Mo	0.0012			ALAP<0.005
Ni	0.021			ALAP<0.005
Cu	0.0038			ALAP<0.005
Al	0.008			ALAP < 0.01
Co	0.005			ALAP<0.005
Sn	< 0.005			As + Sn + Sb
				+ Zr < 0.05
As	< 0.005			As + Sn + Sb
				+ Zr < 0.05
В	< 0.0010			ALAP<0.001

^a ALAP – as low as possible.

Several diameters and lengths of tubes are included. The steel will not only be used for testing purposes, but mock-ups and in-pile blanket sub-modules [4] will be fabricated from material of this batch.

Powder production has started to produce powder for hot isostatic pressing (HIP) of pure EUROFER97 powder parts. In addition, the powder will be mechanically alloyed with oxides to provide testing of ODS EUROFER97. Both in Japan and the EU, other smallscale batches of ODS steels are being delivered for primary testing purposes. In most cases, Y_2O_3 is used for dispersion strengthening, but some TiO₂ particles are used as well to levels of 1% weight.

Besides these large quantities, smaller-size RAFM batches with promising compositions are also being produced. The test results of these small batches will serve to adjust the specifications for the next large RAFM steel batches.

3.2. Manufacturing

Fusion welding is considered to be an essential manufacturing practice. Although other methods might be used to circumvent constraints, most components have fusion welds by necessity. Tungsten inert gas (TIG) and electron-beam welded (EBW) joints of RAFM steels have been recently subjected to mechanical testing. Van Osch et al. reported [5] on the tensile properties of TIG-and EB-welded F82H irradiated up to 2.5 dpa at 575 K. Nishimura et al. [6] reported on the fracture toughness of TIG-welded JLF-1. Both reported hardening in the weld metal after irradiation. The fracture toughness of the JLF-1 welds shows high values (over 400 kJ m⁻²) if the weld is homogeneous and completely defect-free. Defects cause a big pop-in at low toughness, Fig. 1.

It is foreseen that welding of RAFM steel to conventional steel elsewhere in a plant is unavoidable. Test results given by Fontes et al. in [7] show that sound



Fig. 1. J-R curves of different TIG-welded samples [6].

welds between RAFM and Type 316 steel can be made with acceptable properties.

Where shapes are complex, welding cannot always be applied. For such structures HIPping of steel powder can be a good solution. The properties of the resulting structures depend to a large extent on the surface quality of the powder and the HIP temperature and pressure cycle. Progress has been made in the demonstration of complex parts providing samples with acceptable properties [7].

The joining of component parts by HIP processes is a production route with many advantages, but its primary drawback is the high temperatures required by the process. Hishinuma [5] showed promising results for the spark plasma sintering (SPS) process that uses temperatures not in excess of 1200 K. This limit is 100–200 K lower than the temperatures normal for HIP bonding.

It is foreseen that property gradients could be a solution for several design requirements. An interesting example is the idea of bonding plates of ODS RAFM steels onto RAFM components in order to provide sufficient creep strength to the blanket zone near the plasma, the elevated-temperature location where it is most needed. Other approaches could be the preparation of parts with a gradient in the metal powder composition prior to the HIP cycle. These solutions need to be pursued in close co-operation with designers to shortcut development time, improve effectiveness and lower the cost.

4. Characterization

4.1. Mechanical properties

The toughness of RAFM steels, especially after irradiation, is one of the great concerns for the application of this class of steel. Recently, Rieth et al. [8] published results which showed effects of helium content on the DBTT. The precise role of helium in a fusion reactor environment has not been resolved. Irradiations in mixed spectrum reactors generate helium from the boron in the steel at a quite different rate than in a real fusion blanket. Rowcliffe et al. [9] have reported that the reduction in fracture toughness of several FM steels does not correlate very well with the reduction in strain hardening capacity observed in tensile tests. Nevertheless, the results show that an RAFM steel such as F82H has a toughness after irradiation to 2.5 dpa well over that of more conventional compositions. Hamilton et al. [10] observed that for a dose level of 67 dpa RAFM steels have higher absorbed energies in an impact toughness test than conventional FM steels.

Fatigue properties of base metal and weldments of JLF-1 have been compared by Kohyama et al. [5]. The major conclusion is that the fatigue strength and thus



Fig. 2. LCF properties of base metal and weld Kohyama in [5].

the fatigue life of TIG and EB weldments is longer than for the base metal, Fig. 2. Another finding is that miniature fatigue specimens show only a limited deviation from normalized values obtained from large specimens. Ishii et al. [11] performed low-cycle fatigue tests with tension hold times and observed that tension holding reduced the fatigue endurance of F82H steel. At test temperatures over 800 K the tension hold effect influenced strongly the number of cycles to failure.

Petersen [12] has reported thermal fatigue test results on F82H, OPTIFER and the more conventional FM steel MANET. The F82H showed lower thermal fatigue endurance than the MANET steel, whereas OPTIFER had fatigue properties superior to those of MANET. Petersen also observed a detrimental effect of dwell times on the thermal fatigue endurance. He showed that the final thermal–mechanical treatment can control the fatigue behaviour to a certain degree.

Schaefer [7] reported on the preparations to be taken for solid HIP of large components to have acceptable toughness and strength. Both surface quality and dimensions need to be under close control.

4.2. Helium effects

In a fusion environment helium forms in RAFM steel from nuclear reactions with 14 MeV neutrons. Nagasaka et al. [13] have studied the effects of 1000 appm helium implanted in pure 9 Cr steels. The pure steels showed a finer cavity distribution than the more conventional steels. The denuded zone near a grain boundary in clean steels is also narrower than in a conventional steel. Obviously, the lack of impurities affects the cavity formation mechanism.

Schaublin reports [5] that at displacement levels below 0.5 dpa in F82H there is no difference in microstructural defects between as-received, deformed and



Fig. 3. The measured DBTT plotted versus the helium content of the samples, Materna-Morris in [5].

irradiated condition. After 2.5 dpa neutron irradiation at 525 K, the F82H steel shows a high density (3×10^{21}) m^{-3}) of black dots (loops) but no cavities. TEM of the OPTIMAX steel, on the other hand, shows no visible defects, but a faceted cavity density of 2×10^{20} m⁻³ is observed. Shiba has shown [5] that after neutron irradiations up to 57 dpa no cavities are observed in F82H, with one exception. If the F82H is doped with 10 boron to form 320 appm helium, cavities are observed at a density of 2×10^{21} m⁻³. Materna-Morris has observed helium bubbles in several RAFM steels. F82H seems to give the highest values of 4×10^{22} m⁻³ at about 500 K, the same temperature as in the previous studies. Kimura [5], Materna-Morris and others have shown that the mechanical properties (tensile, impact toughness and DBTTT) depend on helium content and temperature in the range of 400-700 K, e.g., Fig. 3. Kimura has also shown that helium affects defect cluster formation in terms of the density and diameter of the defects observed.

It is evident that the RAFM steels respond differently to low-dose irradiations, not only in cavity densities, but also in homogeneity observed in the microstructure. Explanations are not yet complete.

4.3. Ageing effects

In Japan and the EU, ageing treatments of F82H and batches of RAFM laboratory heats have proceeded to the 20 000 h level. The majority of available data is for exposure times up to 10 000 h. Both Alamo [14] and Shiba [5] conclude that the effects up to an 800 K ageing temperature are limited. The microstructures are stable



Fig. 4. Absorbed impact energy of F82H after 10000 h of ageing at indicated temperatures, Shiba in [5].

and so is the response to mechanical testing. The impact toughness decreases considerably, as shown by Shiba for the reduction in DBTT for F82H aged to 10000 h at temperatures above 800 K, Fig. 4, in agreement with the finding of Schaefer and Schirra [15]. Schirra et al. [5] did not observe an influence of thermal ageing up to 20000 h on the 1% yield limit and creep rupture of F82H.

The starting metallurgical conditions of the RAFM heats are generally a normalization temperature between 1300–1330 K and a tempering temperature of about 1020–1050 K. These heat treatment conditions are recommended based on earlier optimalization experiments. Fernandez et al. [14] have performed low-cycle fatigue tests on F82H aged for 5000 h. The results show an increase in fatigue strength after ageing that is not negligible.

4.4. Microstructure

Alamo et al. have reported on the physical metallurgy of Ta-bearing RAFM steels [16]. The major findings are that austenitization temperature and austenite grain size depend on each other through the availability of Ta and N. This is shown in Fig. 5, where it is seen that Ta limits the austenitic grain size; 1325 K is the upper limit for the austenitization temperature. Alamo et al. recommend on the basis of ample experimental evidence that a tempering temperature in excess of 975 K be selected. Hasegawa et al. [17] conclude that Ta-bearing steels such as JLF-1 have a higher creep strength and toughness as compared to steels alloyed with Nb. They observe that the thermal–mechanical treatment has an important influence as well.

Klueh [18] offers an explanation of the beneficial effects of Ta on the impact properties of Ta-bearing steels after irradiation. The reasoning is that with sufficient Ta available the Ta in solution increases the cleavage stress or affects the temperature relationship of the flow stress advantageously. It was also observed that the impact



Fig. 5. The effect of Ta content on austenite grain size [15].

properties deteriorated with dose and increasing irradiation temperature, indicating that Ta might be lost from solution by precipitation during irradiation. This reasoning has to be supported by more detailed electron microscopy.

Coppola et al. [19] have reported their microstructural stability studies with small-angle neutron scattering (SANS) in order to prepare for such studies of irradiated RAFM steels. Their SANS experiments on F82H, supported by extensive TEM studies, indicate the formation of fine precipitation at 825 K and delta ferrite formation above 1100 K. Preliminary experiments on irradiated F82H show promise for the successful application of SANS for RAFM steels.

Pilloni et al. [20] have shown that Ti-bearing steels have thermodynamic properties similar to those of otherwise stabilized RAFM steels. They report that austenite grain size can be very well controlled to obtain the required toughness.

5. Future of RAFM steel development

5.1. Material composition

The production of RAFM steels with low impurity levels has been demonstrated. Technically, improvements in impurity reductions are possible, as recently reported by Lyakishev [21]. Activity reduction to the hands-on level might be reached but requires reducing the concentration of some elements by second orders of magnitude. The optimum depends on the rules that are developed for the recycling of materials and the storage costs. The neutron spectrum is another parameter that determines the level of activation. Considering the experience accumulated, the technical and economic factors of steelmaking do not seem to be the limiting factor in the low activation requirements. Isotopic tailoring remains a technical possibility, but at present it is unlikely to be used on a large scale, because the cost is prohibitive and present recycling schemes seem adequate to solve potential waste problems.

The chemical compositions of RAFM steel will be adjusted as the experience with this alloy class grows. The final Ta level will depend more on limiting radiation effects on toughness than on grain size control, although the two may be connected.

5.2. Manufacturing

Fusion welding of RAFM steels has been demonstrated to produce welds of acceptable quality. Standard laser, TIG and EB weldments have been successfully made and proven to have only defects of acceptable size.

Non-standard welding positions and multiple postweld heat treatment effects in complex welded shapes require additional attention to determine the limits.

The solid HIP joining technique requires rather high temperatures in comparison to other approaches, such as the SPS joining. The latter will need more R&D to assure its position as a versatile joining technique.

Impurity control of steel powder for the HIP process is not yet completely satisfactory. Ideal conditions have been shown for laboratory batches, but demonstrations with large quantities are still lacking for RAFM steel.

The attractive properties of ODS steels are not needed in the complete blanket structure. Cladding structures with ODS steel where it is needed is one practical solution to overcome the fusion welding limitations of ODS steel. Another way to realize components with graded properties is mechanical alloying with steel powder of different compositions, but here the production route is more complicated. Analytical tools to guide the powder HIP technology shape and fill selection are under development. Such analyses greatly reduce the number of trial pressings to verify the chosen geometries. Contributions to this conference indicate the interest and potential of the ODS steels. The behaviour of conventional ODS steel in Phenix as reported by Dubuisson et al. [22] justifies the efforts to develop this category of steels for fusion applications.

5.3. Experiments

The new heats of RAFM steel will not only be exposed in material test reactors with a mixed neutron spectrum, but also in fast reactors. This allows an insight into the difference in helium generation rate of the two reactor types, which could help resolve the uncertainty of the effect of simultaneous introduction of displacement damage and helium on cavity formation and subsequently on mechanical properties. The use of fast reactors, BOR-60 for example, does not mean high-temperature irradiations: a temperature below 600 K

seems to be a feasible irradiation temperature in BOR-60.

Another source of useful information on the development path of RAFM steels are the in-pile irradiations foreseen in the EU and Japan of the so-called blanket sub-modules. These are sections of blankets in configurations that are representative for studies on the behaviour of structural material, breeding and multiplying materials in the HCPB WCLL integrated tests in-pile, Fig. 6. The sub-module will provide data on blanket behaviour, but also on the RAFM steel under mechanical and chemical interactions with the breeding and multiplying materials in tritium. The integrated test might also guide adjustments of the chemical composition and heat treatment of the steel and its weldments. Of course, the damage levels do not reach blanket end of life (EOL) conditions in a mixed spectrum reactor needed to simulate tritium generation - but they certainly will reveal early unforeseen effects, if any.

The use of a 14 MeV neutron source is the ideal test bed for RAFM steel exposure to EOL irradiation conditions that are most realistic. These exposures must answer unresolved questions regarding simultaneous production of helium, tritium and displacement damage at high rates and levels. The sub-module tests are helpful to explore unforeseen interaction effects, but by far the most realistic simulation environment is provided by a 14 MeV neutron source, which is essential for structural materials, and breeding materials, tests alike. The



Fig. 6. Lay-out of an in-pile test of an HCPB-type blanket section constructed with EUROFER97 [4].

development of RAFM steel will in several years advance to a stage that requires confirmation experiments in a 14 MeV source like the International Fusion Materials Irradiation Facility (IFMIF).

5.4. Modelling

In the US, the future emphasis will be more strongly on the knowledge base needed for economically and environmentally attractive fusion energy. For the RAFM development this goal is translating into an extended modelling effort. Both the advanced RAFM steels and their ODS counterpart are part of the effort in the US. The design window limits for these classes of alloys should be established in this way. Major challenges that are considered include ferromagnetism effects on the reactor structure, dpa/helium effects, transmutation effects on properties and compatibility.

A typical example of the development is the multiscale modelling of neutron irradiation embrittlement as outlined by Odette [5]. A system that confines molecular dynamics, thermodynamic rate kinetics, dislocation deformation mechanics and local fracture mechanics should lead to the independent physical validation of the engineering databases. This system is not yet complete, but its approach shows promise to provide analytical hardware tools that will become twice as powerful every 1.5 yr in the next decade.

The EU program is more oriented towards the manufacturing of engineering demonstration devices, such as the HCPB and WCLL test modules in ITER and their precursors: the sub-modules in mixed spectrum reactors. RAFM steels are the candidates for these devices. Therefore, irradiation performance and compatibility are of high importance. Further the EU approach requires qualification of fabrication processes and rules for design, fabrication and inspection. In the program, modelling has a place in the project tasks pursuing metallurgical and mechanical characterisation and qualification for DEMO.

The Japanese program is distinguished in three phases, where in the exploratory and optimisation phase much attention is devoted to modelling of the RAFM steel behaviour. Japan continues the development based, on the one hand, on contributions from universities and, on the other hand, on the strong involvement of technology institutes and the manufacturing industry.

6. Conclusion

6.1. Progress

Following the successful production of the industrialscale heat of F82H RAFM steel in Japan, now the heat of EUROFER97 steel has been delivered in industrial quantities in the EU. These accomplishments show that the production of large heats of these steels with low impurity levels is feasible.

The databases to be used by designers for nearplasma components of fusion power plants grow steadily, especially the mechanical properties, data, including irradiation effects. These data will be helpful for power plant studies and the design of blanket submodule testing devices.

Welds made by fusion welding techniques, such as TIG and electron-beam welding, show properties that make the welding techniques applicable for component manufacturing. On the other hand, HIP bonding allows the shaping of components that cannot be made by fusion welding. RAFM steels can be joined defect free by HIP under strict conditions. To avoid the high temperatures needed in certain cases, SPS has been shown to be an attractive alternative.

An explanation for the effect of tantalum on the reduction of neutron irradiation effects on mechanical properties has been offered, but additional work is essential.

The first results of testing small quantities of ODS ferritic/martensitic steels show promise for ODS application in RAFM steels.

6.2. Critical issues

The interaction of helium and displacement damage in RAFM steels is not well understood. The operating mechanism has not revealed itself so far even at the lower (<100 appm, 10 dpa) damage levels. A tool to investigate this phenomenon under fusion power plant relevant conditions, namely a 14 MeV neutron source, is needed. In this way it would be possible to resolve the uncertainties arising from the low damage levels and high damage rates derived from present tools. The weldability of RAFM steel containing helium levels over 10 appm has yet to be demonstrated. That is, procedures for repair or replacement of RAFM components with high helium content in weld seams need further development.

The toughness of RAFM steels has improved considerably over their conventional predecessors. Further reduction of ductile–brittle transition temperatures should be pursued.

The use of reduced activation steels with ODS in large thick-walled components is attractive for higher operating temperatures. Mechanical alloying and manufacturing techniques are not yet established to the required level. The existing ODS experience only forms the basis for the developments needed to satisfy the special requirements for fusion power plant components. Ultimately, the most versatile component might be built from both pure RAFM steel and only ODS RAFM parts on the hot spots, where their excellent creep strength can be used to advantage.

Commercial fusion power is still a long way off. The major competitors of fusion energy will force strong cost control upon fusion devices. Close interaction of designer and materials technologist for innovative solutions will strongly contribute to the success of fusion power plant penetration in the energy market.

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