Task Title: DEVELOPMENT OF NOVEL REDUCED ACTIVATION MARTENSITIC STEELS WITH IMPROVED CREEP PROPERTIES

INTRODUCTION

A reduced activation martensitic ferritic Fe-9CrWVNbNB alloy designed in a previous stage of the “Fusion” action [1], [2], and manufactured by Aubert et Duval, has been characterised, in terms of microstructure and precipitation kinetics. One of the main traits of this alloy is its reinforcement by MX precipitates, in this case, vanadium nitrides (VN), and by addition of boron. Alongside with the characterisation of the alloy, modelling of precipitation kinetics and microstructure evolution has been performed using MatCalc, a new suite of thermodynamic and kinetic software. MatCalc predictions show good agreement with experimental measurements on phase stability. Using these models it has been possible to design precise heat treatments to obtain microstructures suitable for creep resistance. A comprehensive scheme for the characterisation of the mechanical properties of the alloy after different heat treatments is under way. Early results include those of tensile tests at room and high temperatures and measurement of the brittle-ductile transition temperature, which in both cases show a remarkable behaviour.

2006 ACTIVITIES

Characterisation of MX-strengthened alloy

The characterisation of a MX (VN) reinforced reduced activation martensitic ferritic alloy with improved creep behaviour has been conducted. Microstructural characterisation is complete and mechanical characterisation is underway [5].

The microstructure of this alloy confirms this strategy to obtain improved structural materials for application in fusion reactors and the suitability of this type of alloy for both thin and thick products, due to enhanced hardenability with respect to alloys reinforced with TiC precipitate. The heat treatments designed allow developing a fine distribution of MX (VN) precipitates that remain stable above the service temperature of the alloy and are expected to increase the creep resistance of the alloy without compromising its toughness.

Modelling precipitation kinetics to optimise the microstructure of the alloy

During this work, the characterisation of the cast Fe-9CrWVNbNB has followed a parallel path of microstructural characterisation and thermodynamic and kinetic modelling. MatCalc, a thermodynamics and kinetics package developed by Ernst KOZESCHNIK et al. [3], [4], at the Graz University of Technology (Austria) has been used to model the “as-received” microstructure, and the $M_{23}C_6$ and MX precipitation and dissolution reactions, and to design optimised heat treatments. This package is specially suited for the description of precipitation reactions, allowing the determination of kinetics of reaction, fraction of precipitating phase, composition and size distribution of particles. It is also able to deal with simultaneous precipitation reactions in complex systems composed of many elements and containing multiple phases, like in the present case, where $M_{23}C_6$ and MX precipitation reactions needed to be considered (alongside other phases participating on the precipitation sequence of $M_{23}C_6$).

The phase stability diagram (figure 1) for alloys of a standard Fe-9CrWVNbNB alloy has been calculated using MatCalc.

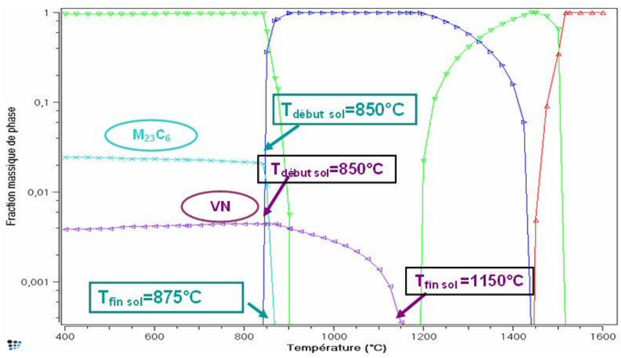

Figure 1: Phase stability diagram of Fe-9CrWVNbNB alloy

The same software also allows to calculate the TTP diagrams (figure 2), (Temperature Time Precipitation, analogous to TTT diagrams).

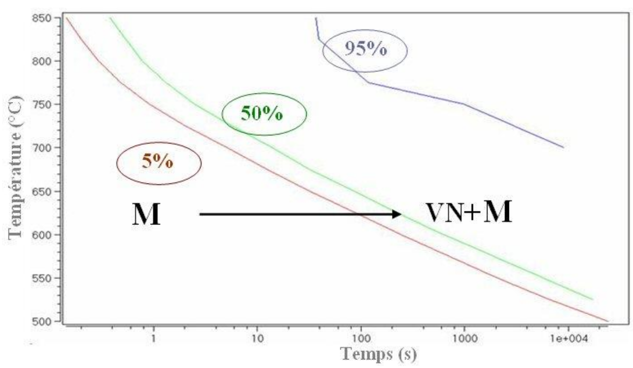

Figure 2: Time Temperature Precipitation of VN phases on Fe-9CrWVNbNB alloy. Percentages represent the fraction of the total precipitate fraction at equilibrium for each given temperature

Using these diagrams, it has been possible to design different heat treatment that produce a microstructure with a fine and homogeneous dispersion of MX precipitates (figure 3).

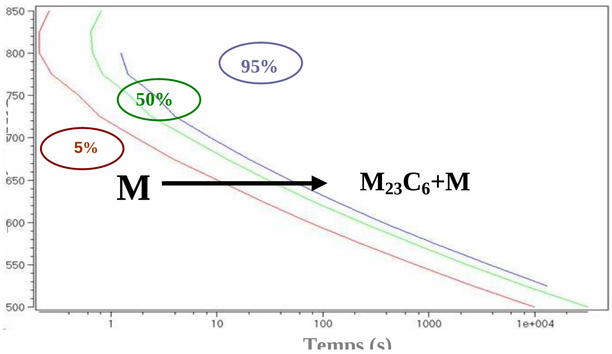

Figure 3: Time Temperature Precipitation of $M_{23}C_6$ phases on Fe-9CrWVNbNB alloy. Percentages represent the fraction of the total precipitate fraction at equilibrium for each given temperature

Armed with these models, it has been possible to suggest two different heat treatments that would allow to produce MX-reinforced microstructures suitable for high temperature applications and creep resistance, as shown in figure 4. Heat treatment of the type a) (named here 'standard') follows a classical approach in which the microstructure is over-aged at a temperature well above service temperature and for a long time (~10h), to ensure that the resulting microstructure will hardly evolve during service. The heat treatment of type b) (named here 'optimised') leads on the other hand to a much finer distribution of reinforcing precipitates, without an excess of over-aging. It consists in a double tempering, where the reinforcing phase is made to precipitate in a heat treatment below service temperature, followed by a short stabilisation (~1h) at a temperature well above service conditions.

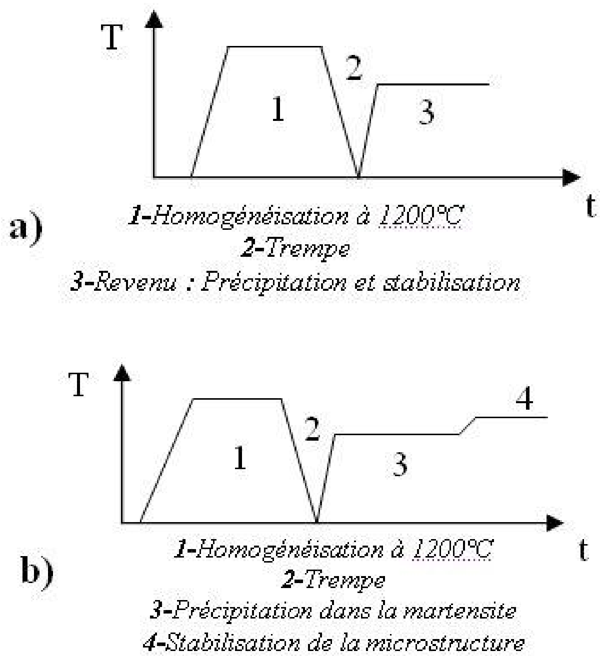

Figure 4: The two different types of heat treatments suggested for Fe-9CrWVNbNB alloy

To summarise, the simulation of heat treatments allows describing the transformation and precipitation reactions occurring during diverse heat treatments. By using a software package like MatCalc, it is possible to determine the mole fraction of precipitate phases, their number density, composition and size distribution.

Preliminary results from the determination of mechanical properties

The brittle-ductile transition temperature has been determined for the Fe9CrWVNbNB alloy with the a) type or 'standard' heat treatment.

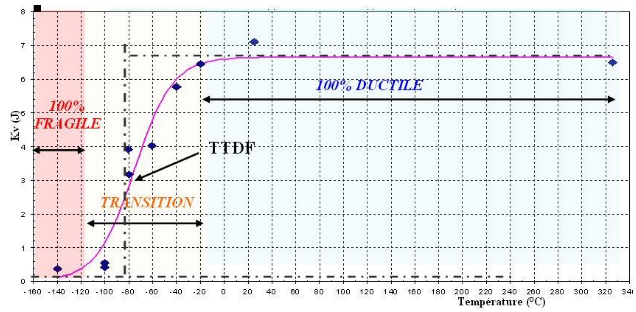

Figure 5: Determination of the brittle-ductile transition temperature on Fe-9CrWVNbNB alloy

Uniaxial tensile tests at room and high temperatures allow to compare the two types of heat treatments between them and to reference alloy PM2000.

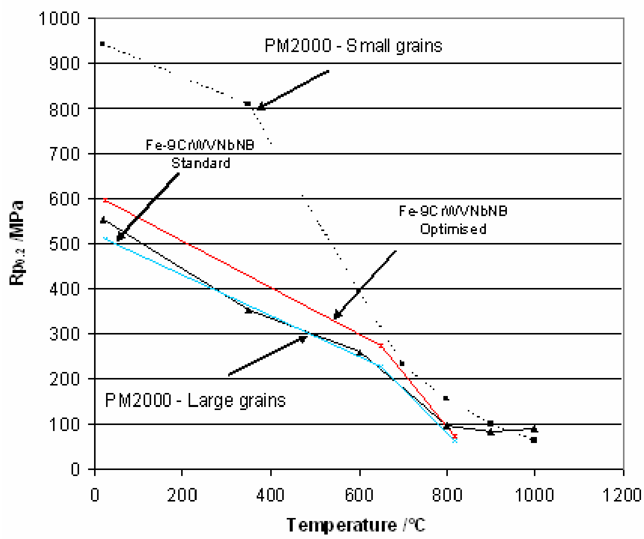

Figure 6: Uniaxial yield strength of Fe-9CrWVNbNB alloy, in two different conditions, compared with PM2000

CONCLUSIONS

A new family of reduced activation Fe-9CrWVNbNB martensitic alloys for fusion reactors has been developed and characterised. This type of alloys benefits on the reinforcing properties of a fine distribution of MX precipitates and a small amount of boron, in addition to $M_{23}C_6$ carbides used traditionally. This type of precipitates are more stable that $M_{23}C_6$ carbides used in alloys of the type EUROFER and others, and therefore the resulting alloy could have improved creep and toughness properties and to be able to work at higher service temperatures than the alloys used presently, up to 650°C for 10,000h.

During the development and characterisation of alloys Fe-9CrWVNbNB, substantial know-how in modelling of the microstructure using advanced thermodynamic, kinetic and statistic models has been put to use to develop two different types of heat treatment for this specific alloy. The models used allow to calculate the precise precipitate distribution of various reinforcing phases, and therefore to design two different heat treatments leading to optimal microstructures.

The alloy presented has been heat treated to both conditions and is presently being characterised in terms of fine microstructure, mechanical properties, toughness and creep resistance.

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Task Title: MICROSTRUCTURAL EVOLUTION OF Fe-C MODEL ALLOY AND EUROFER UNDER 1 MeV ELECTRON IRRADIATION WITH AND WITHOUT He PRE-IMPLANTATION

INTRODUCTION

A multiscale modelling programme has been initiated whose ultimate aim is to study the radiation effects in the Eurofer ferritic/martensitic steel in the presence of high concentrations of nuclear derived impurities [1]. The development of these models requires an experimental validation and, in the case of complex multicomponent industrial alloys such as Eurofer, the values of model parameters will have to be tuned based on experimental data. Although many microstructural investigations following irradiation experiments of steels, including Eurofer, have been carried out in the past, the data are not adequate for model validation because the irradiation conditions are often complex and/or not well known. We have therefore proposed to perform parametric irradiation experiments of a model Fe-C alloy and Eurofer, under well controlled conditions (temperature, dose, damage rate), using 1 MeV electrons with and without helium pre-implantation. 1 MeV electrons create only isolated defects (Frenkel pairs) in steels, i.e. the primary damage is perfectly well known, which is an additional advantage in view of the comparison with model predictions.

2006 ACTIVITIES

The workprogramme initially forseen for 2006 consisted of 1MeV electron irradiations of Eurofer following pre-implantation with helium. Helium implantation experiments of Eurofer specimens, in the ferritic metallurgical condition (see the 2005 Fusion Technology report for the details of the heat treatment applied in order to obtain a ferritic microstructure) were therefore carried out at room temperature, using the Irma accelerator of CSNSM (CNRS, Orsay University). A total fluence of $10^{14}/cm^2$ ^4He particles of 10 keV energy were implanted in discs 3 mm in diameter and 100 µm in thickness, which had been electropolished beforehand on the side facing the ion beam. The average implanted helium concentration over the total range of the ^4He particles was about 50 appm, with a concentration profile shown on figure 1.

The next step should have been to electropolish the discs on the backside, using a protective lacquer on the helium implanted front size, in order to prepare specimens for subsequent irradiation with 1 MeV electrons. However, as already mentioned in the 2005 report, difficulties were encountered with the use of the High Voltage Electron Microscope (HVEM): in particular, the vacuum quality required to perform irradiations while heating the specimens could not be achieved in the microscope column.

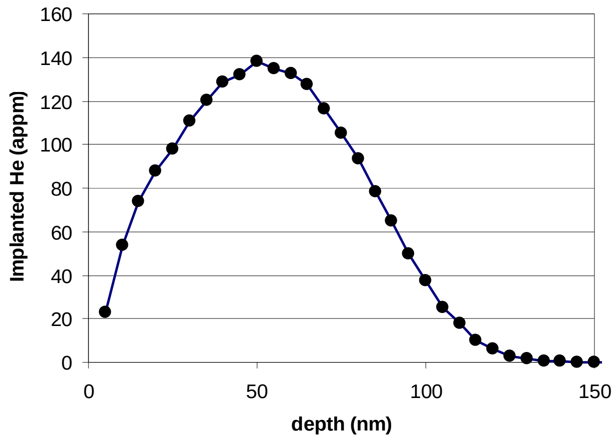

Figure 1: Helium concentration profile as a function of depth in the material for Eurofer disc specimens implanted with 10 keV ^4He particles

Despite efforts to solve this problem, a satisfactory solution was not found. As a result, and taking into account the age of the microscope (21 years), a major maintenance/upgrading operation of the microscope was envisaged. As a first step, a feasibility study of this operation was carried out, with the help of a specialized company from the UK (A&J Scientific Limited). The main conclusion of the study was that this operation is both feasible and worthwhile and could be implemented over a three-year period [2]. It would include major modifications of both the vacuum and high voltage systems as well as fitting a digital camera system. In addition, modifications would be implemented in such a way that the microscope would be part of the time available for experiments after the first year of the upgrading operation.

CONCLUSIONS

He implantation experiments of Eurofer specimens were conducted using ^4He particles of 10 keV energy up to an average concentration of 50 appm Helium. However, the subsequent 1MeV electron irradiations could not be carried out due to operating problems with the HVEM.

It was therefore decided, following a feasibility study, to carry out a major upgrading operation of the microscope in order to be able to perform the planned electron irradiations.

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