IV SIMPÓSIO NIPO-BRASILEIRO DE CIÊNCIA E TECNOLOGIA Área 10: Aplicação de Radiação e Radioisótopos

LIGTH IONS CYCLOTRON BOMBARDMENT TO SIMULATE FAST NEUTRON RADIATION DAMAGE IN NUCLEAR MATERIALS.

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# ABSTRACT

In this work the applicability and limitations of the use of Cyclotron light ions bombardment to simulate the effects of the neutron irradiation are presented. Light ions with energies of about 10 MeV are capable to produce homogeneous damage in specimens suitable for measuring bulk mechanical properties although their low damage rate of 10<sup>-5</sup>dpa.sec<sup>-1</sup> limit the dose range to a few dpa. On the other hand Cyclotron alfa particle implantation provides a fast and convenient way of introducing Helium with a minimum of side effects so that we can take advantage of this technique to get better understanding of the mechanisms by which this insoluble gas produces high temperature embrittlement.

Some experimental details such as dimensions and cooling techniques are described. Finally a description of the infrastructure for Cyclotron alfa particle implantation and a Creep-test facility of the Division of Radiation Damage at IPEN-CNEN/SP are presented.

### 1. Introduction

Since the discovery made by Cawthorne and Fulton, that stainless steel used as fuel cladding in a fast reactor, presents the void swelling phenomena; a great deal of effort has been made to develop simulation techniques using ion beams to make accelerated studies of void swelling and other radiation effects that occur in structural materials under irradiation. The use of ion beams to study void swelling was introduced by Nelson and Mazey at Harwell and since then this techniques has been use extensively and with success in other leading material research laboratories. At IPEN-CNEN/SP, with the technical assistence of the Institute of Solid State Physics of KFA-Julich, FRG, we have built an infrastructure to study He - embritlement problems in metals using the compact cyclotron for  $\alpha$ -particle implantation.

In this paper, a brief introduction about radiation damage is out-lined followed by a discussion of the applicability and limitation of the use of light ion bombardment to simulated neutron irradiation effects. Some experimental details such as, specimens dimensions and cooling techniques are presented.

## 2. Radiation damage in metals

As it is well established the macroscopically observed radiation damage phenomena is a consequence of two basic interaction between the irradiation particle and the lattice atoms of the target material (Fig.1).

a) The irradiation particle transfers to the lattice atoms recoil energy by elastic and inelastic collisions and when

this energy is greater than the threshold energy (~ 40 eV) a displacement of atoms from its lattice site occur. In the specific case of fast neutron which transfer energies in the 10 KeV - range, the primary recoil atom is not only displaced itself but also is capable to displace other atoms leaving vacant lattice sites. The result of this collision process is the formation of Frenkel-defects (interstitial - vacancy) which, immediately after collision are distributed in displacement cascades formed by a nucleus of vacancies surrounded by interstitials. In case of collision with other irradiation particles (e,ions) the induced defects will be distributed in isolated Frenkel-pairs or in small cascades.

b) Hight energetic particles can cause nuclear reactions creating foreign elements in the target material. The inert gas He produced by  $(n,\alpha)$ -reaction plays an important role in the loss of ductility of irradiated metals.

The fate of the defects induced by irradiation will depend on the temperature range in which the irradiated material will stand since, interstitials and vacancies are highly mobile at normal temperatures and they will interact with other defects when diffusing through the crystal lattice. The most frequent event in defect interaction is the recombination process where an interstitial and a vacancy meet, thus recovering the lattice structure, but when defects of the same kind meet, the formation of clusters take place. Beside this cluster process some defect can be trapped by crystal defects, such as dislocations, grain boundaries and precipitates. Under certain temperature conditions and high irradiation doses a threedimensional agglomation of vacancies (voids) are formed. The formation of voids has been extensively studied after it became clear that such defects is the cause of swelling. Helium is practically insoluble in solids (3) and, at ele vated temperatures tend to precipitate in form of He filled bubbles, normally near grain boundaries, precipitates and dislocations. The bubbles in the grain boundaries play an important role in the embrittlement of metals. From the many properties changes induced by irradiation, swelling, in-pile creep and He-embrittlement are of special importance in reactor technology.

# 3. Simulation experiments

The investigation of mechanical properties changes in structural components using material test reactors require sofisticated techniques associated to technical difficulties, high cost and long periods of time. On the other hand, reactor irradiations are an absolute necessity, since, reactor components must be tested under realistic environmental conditions. For basic research which aims at fundamental understanding of radiation damage and as well as a tool to screen structural materials, simulation techniques in which the neutron radiation effect is simulated by high energy charged particle bombardment are a promising approach.

Heavy ions and electrons from HVEM produce high displacement rates ( $10^{-3}$  to  $10^{-1}$  dpa/sec) (Fig.2) compared with rates obtained with fast neutrons ( $\sim 10^{-6}$  dpa/sec) but, on the other hand, the damage range is less than 1  $\mu$ m, that is only suitable for TEM observations. To study machanical properties changes like, stress-strain, creep, fatigue, etc, the specimens to be tested required a certain thickness of around 25  $\mu$ m<sup>(4)</sup> in order to obtained results comparable to those obtained with bulk samples.

Light ions (p,d, &) with energies in the range of 10 - 30 MeV (compact cyclotron) produce uniform damage in thicker samples, suitable for mechanical tests. Unfortunately the increase in damage depht is achieved with high reduction in damage rate. However, simulation with light ions bring still some advantages such as high experimental reproducibility, due to better control of irradiation parameters and teste parameters (temperature, stress

and test environment) and because, cyclotron experiments have a high duty factor. These points together with somewhat higher damage rates bring integrated advantage in time and cost. Another disandvantage, besides sample thickness limitation, is the deffect structure ob - tained with charged particles and with neutrons.

### 4. Experimental considerations

# a) Sample dimensions

The range of the bombarding ions determine the maximum specimen thickness that, in simulation experiments, is the critical dimension. Since the displacement rate of changed particles decreases with increasing energy (Fig.3), high current densities are used up to a point that the removal of the irradiation heat leaves the damage rate in values greater than those obtained in fast reactors. The use of thin specimens to measure mechanical properties arises the question whether the result obtained are comparable with those in bulk samples. Experiments in stainless stee show that the tensil behaviour of foil samples match to those of bulk material as long as their thickness exceeds about three grain diameters (Fig.4).Results from other materials imply a practical specimen thickness of around 25 µm.

b) Cooling techniques and temperature control The interaction of light ions with target mate rial produces heat rates of 2 to 20 kW/cm $^3$ . The removal of such energy densities are possible from foil specimens when they are attached to a temperature control heat sink. This cooling technique is used mainly in  $\alpha$ -implantation experiments (Fig.5).For "in beam" experiments where the temperature is a critical parameter, cooling systems using mainly, Helium gas in turbulent flow (100 m/sec) were able to achieve cooling rates of 50 W per cm $^2$  (6). In creep experiment, the temperature control has to achieve variations of  $\pm$  0,5 K in order to measure creep rates without the interference

of specimen thermal expansion. In Fig. 6, an experimental arrangement is shown, to study irradiation-induced creep with a cyclotron. The main features of this arrangement are the He-gas cooling system and the compensated heating rate obtained by a direct current through the foil.

5. Infrastructure for  $\alpha$ -particle implantation and post-irradiation creep measurements.

Fig. 5, shows the experiments arrangements for ∝-particle implantation. A uniform He implantation, in the whole specimen volume, is obtained by means of an energy degrader system formed by a rotating wheel containing Al-foils of different thicknesses. The specimens to be implanted are direct soldered on a water cooled Cu-block and the concentration of the implanted He is calculated from the integrated current that hits this thermal sink. Some typical values obtained in an implantation process are also presented in Fig. 5. The experimental facility for post-irradiation creep test is presented in Fig. 7. All parts of the suspention system are made of Invar to give thermal stability. The creep strain is measured by a LVDT strain gauge with a resolution of  $5 \times 10^{-5}$  cm/cm, and the load is apply by means of a spring whose tension can be varied with an stop edge nut. The critical creep test temperature is provided by an electric furnace couple to a PID-temperature regulator. Such arrangement temperature variation of - 1°C is obtained during the whole creep test.

Previous creep-measurements in stainless steels has shown that this creep test facility have a high degree of thermal and mechanical stability suitable for precision creep measurements.

#### References

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# Figure capture

- The interaction of high energy neutrons with the nuclei of a metal lattice.
- Maximum attainable displacement rate for different bombarding particles as a function of the distance into Nickel.
- 3. Displacement rates per particles \( \mu \text{A/cm}^2 \) current density for charged particles in Nickel.
- 4. Tensile tests at room temperature in a Hall-Petch plot.
- 5. Experimental arrangement for  $\alpha$ -particle implanta tion into foil specimens.
- Experimental arrangement for irradiation-induced creep studies using cyclotron.
- 7. Experimental facility for creep test with foil specimens.

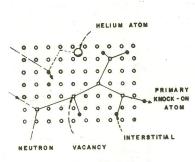
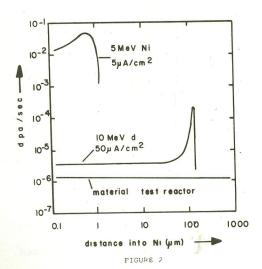
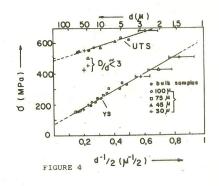


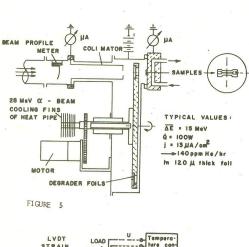
FIGURE 1

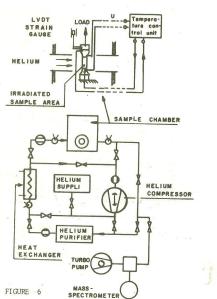


10-6
2
2
3
10-7
2
10-8
1 2 5 10 20 50 100

FIGURE 3 Ε (MeV)







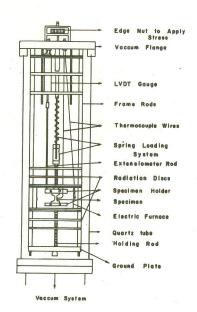


FIGURE 7