RADIATION DAMAGE STUDIES IN METALS AND ALLOYS- THE BRAZILIAN EXPERIENCE.

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1. INTRODUCTION

Reactor components are exposed to high fluences of energetic particles which produce changes in their atomic structure. These desturbances, consequently, influence the electrical, magnetic and mainly mechanical properties (e. g. hardness, strenghth, ductility and dimensional stability) which are the most important characteristics for practical application of metals and alloys. Whereas some changes may be considered as "beneficial" effects (1),(2) in metals and alloys, most of them are destructive justifyng the expression " Radiation Damage" for the influence of radiation on materials.

Considering the irreversible trend trowards the development of nuclear technology and uses of nuclear energy, it becomes evident that Radiation Damage in reactor components must be seriously considered for a safe and economic operation of present fission reactors, and mainly, on fusion reactor of the future. Therefore it is essential to acquire a fundamental understanding of radiation damage effects and develop radiation-resistant materials.

An interesting comparison cam be made on the vulnerability of metals and other materials, by the folowing example: the core components of a fast breeder reactor are exposed to a fast neutron flux of approximately 10^{15} n/ cm^2. sec., which results in about 300 displacements per atom (dpa) after 10 years of operation, practically destroyng the original structure. Another irradiation time cumulative effect is the swelling in present reactor stainless steels, which is a technologically unacceptable disturbance. Plastics desintegrate at much lower doses ($\sim 3.10^{-4}$dpa), and 10^{-12} part of the above mentioned dose would develop pathological processes in bio-tissue leading to the destruction of a living organism. The catastrophic effect of radiation on organic material is due to the fact that a replacement of an atom by another element alters the chemical nature of the material. By contrast, a random exchange of atoms between different lattice sites has no macroscopic effect on pure metal and disordered alloy for relatively small doses, since an appreciable part of the absorbed radiation energy goes into electronic excitation and ionisation, and does not produce structural changes in metallic materials, other than heat. This is not the case of ionic conductors, semiconductors and insutators whose physical properties are strongly influenced by electronic excitation.

The radiation damage study consists of two major, not completely overlaping, fields : basic research on pure materials and technological studies (data collection) on commercial reactor materials. Research workers are interested in basic properties of point defects and structure changes, performing experiments at low-temperature, low-dose irradiations using elaborate methods to dermine the atomic behavior of interstitials and vacancies. Very pure metals and single crystals are used as samples with one kind of predominand defect present, in order to obtain reproducible results. On the other hand, nuclear engineering, to design and construct advanced reactors, must face technological challenges like radiation-induced swelling, enhanced embrittlement and creep of commercial alloys, and needs data on the behavior of these materials after several years of reactor service at high doses and temperatures. I the last decade a convergence of both fields has been noted and is due to great advances recently made in dermining the structure and basic properties of point defects in pure metals with the research presently turning to defect aglomerates in dilute and concentrated alloys, and mainly to the development of simulation techniques (charged particle irradiation) used to characterise commercial materials with regard to their radiation resistance. In spite of the convergence of basic and technological researches, many results and their theorical understanding are still confined to model materials of structures much simpler than those of commercial alloys. Therefore, the macroscopic property changes in reactor materials are only qualitatively understood with many open questions regarding detailed mechanisms leading to embrittlement, swelling, radiation-enhanced diffusion, etc., however the ecquired knowledge is sufficient to identify the areas where radiation damage in structural materials can endanger the safe and economic operation of reactors.

Regarding the structural materials requirement, the following considerations must be made. At present, commercially available pow er reactors are of the following three types: (LWR)- light-water reactors, and to a much smaller degree, the (HTR) high- temperature reactors and, (HWR) heavy-water reactors, which rely on the rare U-235 isotope as fuel. For this reason other types of nuclear energy sources are being developed in several countries one of them the liquid-metal fast-breeder reactor (LMFBR), to make available a proven and environmentally safe commercial reactor in the next decade, which would greatly increase the effective uranium resources, since it "burns" both U-238 an U-235 isotopes. Another type of reactor is the controlled thermonuclear fusion reactor whose practical feasibility has not yet been demonstrated, but in last years such progress has been achieved that researchers are strarting to analyse the technological and economic aspects of fusion reactors where material considerations are of paramount importance (3). Since here only irradiation-induced property changes will be discussed, references (4, 5 and 6) are suggested for further informations on different types of reactors.

In boiling an pressurized-water reactors where the core structure-consisting of fuel cladding, pressure tubes, grid plates, control rods,etc - is exposed to fast- neutron fluxes of order of 10^{13} n/cm^2. sec and temperatures around 300°C, the radiation damage normally is not the life-time limiting factor. The most important radiation effects are low-temperature embrittlement, irradiation creep in stainless steel and Zircaloy, and less frequently an increased hydrogen adsorption and corrosion produced by radiolysis.

Due to their high safety relevance, the thermal reactor pressure vessel deserves great attention. The pressure vessels are generally made of low-ferritic steels welded by special processes and, during their life-time, are exposed to fluences in the range of 10^{18} - 10^{19} n/cm^2 (fluxes up to 10^{11} n/cm^2. sec), at temperatures below 300°C. Low-temperature embrittlement may occur, shifting the ductile-brittle transition temperature into the operating range (20 - 300°C) of rector pressure vessels (7) as shown in Fig. 1.

The diminished toughness (8) of the irradiated material may lead to spontaneous crack propagation and fracture of the reactor containment vessel; with catastrophic consequences. Due to this, extensive investigations have been carried out and should proceed intensively in the future to make available a broad data base to allow reliable technological predictions. In parallel, surveillance samples of material are irradiated in the neighborhood of the reactor

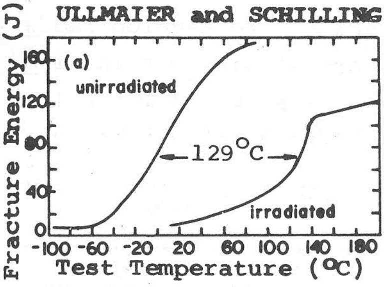

Fig.1 - Ductile to brittle transition shift of Ni-Mo-Cr reactor pressure vessel steel after irradiation with 1.25×10^{20} n/cm^2 (7).

core and tested before the pressure vessel atains the corresponding fluence. If the control of technological problems has been achieved (only apparently), the physical understanding, on atomistic scale, has not yet been quantitatively determined. It is a known fact that the irradiation hardening and embrittlement in pressure-vessel steels is a consequence of point-defects aggregation (vacancies and intersticial atoms), however the role of impurity atoms and other misrostructural defects in stabilizing and affecting these clusters in commercial alloys is far from being completely unterstood.

Gas-cooled high-temperature reactor (HTGR), have concrete vessel , fuel contained in graphite coating, and only a few metallic structural components, reason why no serious radiation damage problems in metallic components are expected, with the exception of control rods. These operate at high temperatures (~950°C) and are exposed to fluences which could cause high-temperature embrittlement. Considerable radiation - damage effects like, volume change, thermal conductivity, and mechanical properties are expected to occur in the graphite coating, but are not discussed in this paper.

In contrast with LWR, the LMFBR operate with, liquid sodium cooling at temperatures up to 700°C, fluxes exceeding 10^{15} n/cm^{2}sec and high mechanical stresses (~100MPa), which lead to material deteriorations that limit the lifetime of many components below economicaly desirable values. The most representative parameters related to radiation damage problems in fast-breeder and fusion reactors, are compiled in Table 1.

The operating temperature of LMFBR is limited by structural material available which is an austenitic stainless steel. The power density level determines the neutron flux, helium production rates, stress levels and fuel expansion. These conditions combined, lead to following destructive effects, in decresing order of deterioration: - swelling by void growth - irradiation creep - high temperature embrittlement and radiation enhanced diffusion. Investiga tions are being caried out, on world-wide scale , to develop radiation-resistant materials whose properties vary within certain limits established by engeneering requirements, as shown in Table 1.

TABLE I ANTICIPATED STRUCTURAL MATERIAL REQUIREMENTS FOR FUTURE REACTOR SYSTEMS (after Ref.(9)).

Parameter	unit	Fast Breeder (stell)	Fusion ractor
Temperature	^{o}C	300-600	300-500 (steel) 500-1000 (refract.)
maximum instantan. displacement rate	dpa/s	$\sim 10^{-6}$	$3X10^{-7}-10^{-4}$(magnetically confined) 1-10(inertially confined)
avarange displacement rate	dpa/year	~50	10-30
Helium production	appm/year	~10	200-600(steel),25-150 (refract.)
No. of power cycles	$year^{-1}$	~10	$10-10^{5}$(magnetically confined) $10^{7}-10^{9}$(intertially confined)
stress level	MPa	60-120	60-200
Maximum permissible:			
Volume change(lifetime)	%	<5	<10
Creep strain(lifetime)	%	<1	<1
Ductility(elongation)	%	<1	<1

In spite of the difference in physical concept of LMFBR and thermonuclear fusion devices, there are some similarities in the radiation damage problems for the structural materials in both systems. In analogy to the core of a LMFBR, the first wall and blanket materials sorrounding the plasma are exposed to high temperatures, intensive mechanical stresses and fast neutron fluxes higher than 10^{14} n/cm^{2}, depending on the wall energy loading which varies from 1 MW/m^{2} to 7 MW/m^{2} for different fusion reactor designs. However, it is already known that the neutron energy spectrum will be considerably higher than that in a fission reactor due to the 14MeV neutrons produced by fusion. These will yield a strongly anhanced Helium production by (n,α) reaction leading, as a consequence, to high - temperature embrittlement.

For the evaluation of the critical radiation damage problems, several simulation techniques have been introduced, with the inicial aim of producing high damage rates for swelling studies after charged particles inplantation. Very high swelling rates were achieved by the implantations with heavy ions (5MeV) - 10^{4} greater than in a typical fast breeder reactor, but with the handicap of a very limited damage range (1 µm) allowing only TEM observations on very thim samples.

Light ions , like p, d and α, with energies from 5 to 30 MeV are suitable for homogeneous damage production in 25 to 100 µm thick samples apropriate for bulk mechanical properties measurement like : creep, fatigue, stress-strain curves, etc., but limited to a reduced damage rate of approximately 10^{-5} dpa/sec (10). With α implantation the highest appHe/dpa rates (~10^{4} appmHe/dpa) can be achieved for simulation studies of He embrittlement in materials (11).

TABLE 2. CHARACTERISTIC FEATURES OF DIFFERENT SIMULATION TECHNIQUES (10)

Particle; energy range;	Advantages	Disadvantages	Main field of application
Heavy ions (self ions); 2-5 MeV Van de Graaf	Extremely high damage rates (100dpa in 3 hours);	Very small ranges (1µm) inhomogeneous damage, surface effects, unknown distribuition of high stresses	TEM investigation of swelling by void formation; screening of swelling resistant alloys
Light ions (p, d,α) 5-30 MeV; cyclotrons	Somewhat higher damage rates than reactors; homogeneous damage in $\cong$100µm thick samples; mechanical tests possible	Limited to 5 dpa; damage structure different from n-damage	Simulation of "in-pile" mechanical properties changes (irradiation creep, fatigue, embrittlement, influence of stress on swelling)
Electrons; 0.2-3 MeV; HVEM	High damage rates; production and observation of defect structure simultaneously	Very thin layers (surface effects) and small areas (stresses); damage structure very different from neutrons (single Frenkel pairs)	In-situ observation of the buildup of damage structure: dislocation loops, voids, cluster complexes, etc.

The high - voltage electron microscopy (HVEM) is especially useful as a simulation technique since permits the observation of structure changes during the radiation damage generation with fast electrons (~1 MeV). Some of the more important simulation techniques are listed on Table 2. In the last decade several laboratories have developed techniques of simulating neutron-damage phenomena through charged - particle bombardment. Although the first Radiation damage experiments date from 1965 the simulation studies started at Radiation Damage Division - IPEN in 1984 on cyclotron α implantations and the results obtained have shown that simulation techniques can provid <u>fast</u>, <u>inexpensive</u> and <u>useful</u> <u>information</u> about many aspects of radiation damage problem. Particularly, basic studies, as well as <u>technological characretization</u> for <u>material development programs</u>

would be unthinkable without the use of simulation techniques.

2. EFFECT OF RADIATION ON CRYSTAL LATTICE

The macroscopically observed radiation damage phenomena are a consequence of two elementary interactions between the bombarding particles and the lattice atoms:

a) Neutrons, ions and electrons with incident energy E transfer a recoil energy T to the atoms; if T > Td (Td-treshold energy) creating a Frenkel pair (vacancy-intersticial)

b) Transmutation nuclear reaction produce considerable concentrations of foreign atoms within the material, such as Helium gas due to (n,α) reaction plying an important role in the mechanical stability under fast-neutron irradiation.

The primary event, when the primary knock-on atom (PKA) is produced, is characterized by the probability of transfering a recoil energy T to the atom, i.e. by the differential cross-section $d\sigma(T,E)$. For ions, $d\sigma/dT$ is given by the Rutherford cross-section-proportional to $(ET^2)^{-1}$ - showing that small energy transfers are favored. Fast neutrons have a contrasting behavior with $d\sigma/dT$ almost constant for small (10^2 eV) and medium (10^4 eV). The maximum transferable energy, for non relativistic particles is given by:

$$T\ max = \frac{4\ M\ m}{(M + m)^2} \quad (1)$$

here: M - mass of lattice atom

m - mass of the incident (bombarding) particle

The fundamental problem of Radiation Damage can be summarized by the following equation:

$$C_d = \int_0^{E_{max}} \int_0^{T_{max}} v(T)\ \frac{d\sigma(T,E)}{dT}\ \frac{d\phi(E)}{dE}\ dT\ dE = \sigma_d \cdot \phi \quad (2)$$

where: C_d - displacement rate

v - damage, function (averange number of atomic displacements produced per PKA with recoil energy T - is zero for $T < T_d$.

ϕ - fluence (integrated flux)

σ_d - displacement cross-section

The comparison of damage structures - cascade size distribuiti on due to different incident particles - can be made by means of eq. (3):

$$C'_d = 1/\sigma_d \, \phi \int_0^{T'} \int_0^{E\,max} v\,(T)\; d\sigma\,(E,T)\; d\,\phi\,(E) \qquad (3)$$

where: C'_d - fraction of displacements produced by primary recoil atoms with $T < T'$

C'_d is shown in Fig. 2 for diferent bombarding particles in Ni. The recoil energies for which, e.g., half of the atomic displacements are produced, are:

~50 e V for 1 MeV electrons
~ 5 KeV for 13 MeV α-particles
50 KeV for 5 MeV Ni - ions
~100 KeV for 1 MeV neutrons

The physical meaning of this curves (Fig. 2) can be explained as follows: the steepness of the curves indicates the energy range of recoils with siginificant contribuition to the damage: For exemple:

- fast neutrons produce recoils in a narrow energy range-between 10^4 and 10^5 eV - meaning that the majority of Frenkel pairs is distributed in large cascades.
- α - particles, have all recoil energies - from 100 eV to 1 MeV contributing equally to the damage yielding a broad spectrum of cascades containing a variable number (up to thousands) of defects.

3 - RADIATION DAMAGE STUDIES AT THE INSTITUTO DE PESQUISAS ENERGÉTICAS E NUCLEARES - IPEN-CNEN/SP.

Radiation Damage research started at IPEN-CNEN/SP in 1965 as a consequence of technical cooperation between Brasil and France, in ~~a~~ group denominated GRESIL (short for Grenoble-Brasil). In 1970 the group grew to a departamental status - Materials Science and Technology Dept-under the management of Prof. S. Watanabe. In 1976 the Radiation Damage Division was created, under the author's supervision performing the radiation Damage Studies and compact cyclotron instal

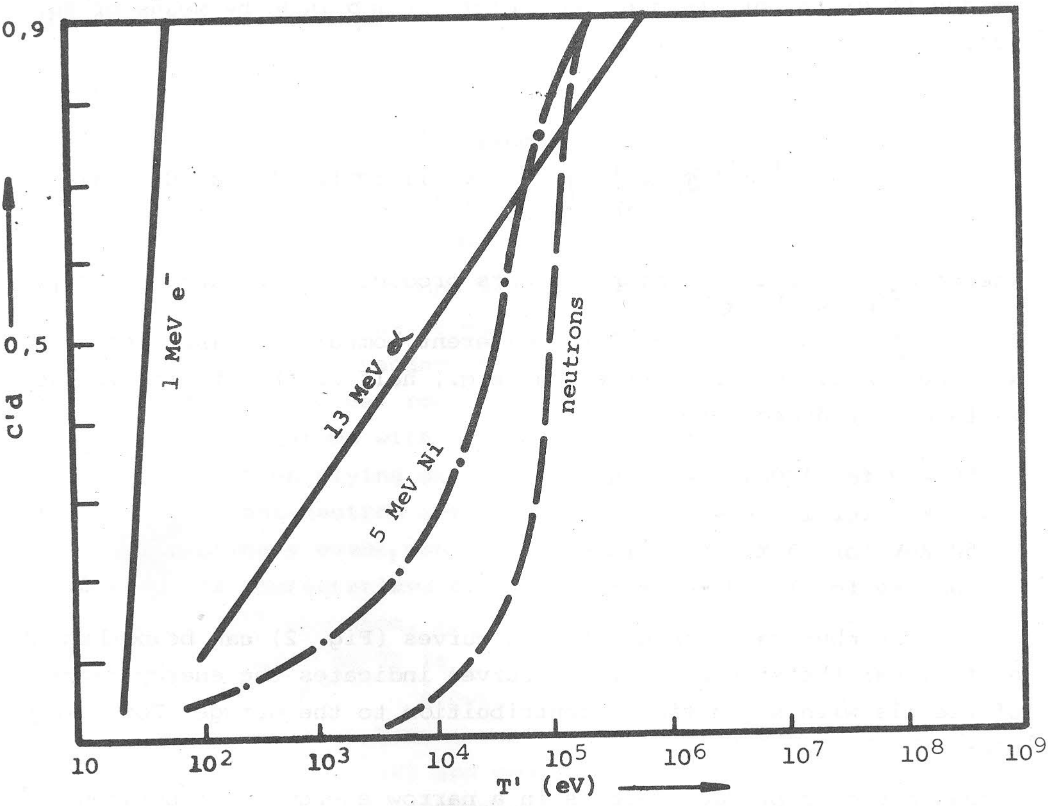

Fig.2. Function C'_d calculated for Ni irradiated by different particles (10).

lation and operation. In the last 22 years the radiation damage group produced several publications (12 - 72) and M. Sc. and Ph. D theses. In what follows, only the most important experiments will be mentioned. In 1965 the first work was performed an order- disorder transformation on Cu - Pd (50-50 at%) and FeNi Mo (49-49-2%) using an "in-situ" irradiation device (Fig.3) which allowed the study of isothermal kinetics under enhanced diffusion conditions, at high temperatures and controlled atmosphere by means of electrical resistivity.

3.1 - Electrical Resistivity

Cu Pd and FeNi alloys showed different behaviour under irradiation (13). While Cu Pd had almost coincident relaxation curves (Fig. 4), FeNi (Fig. 5) presented distinguinshable short and long range order with the critical order-disorder transition at $T_c=515\pm5$ °C.

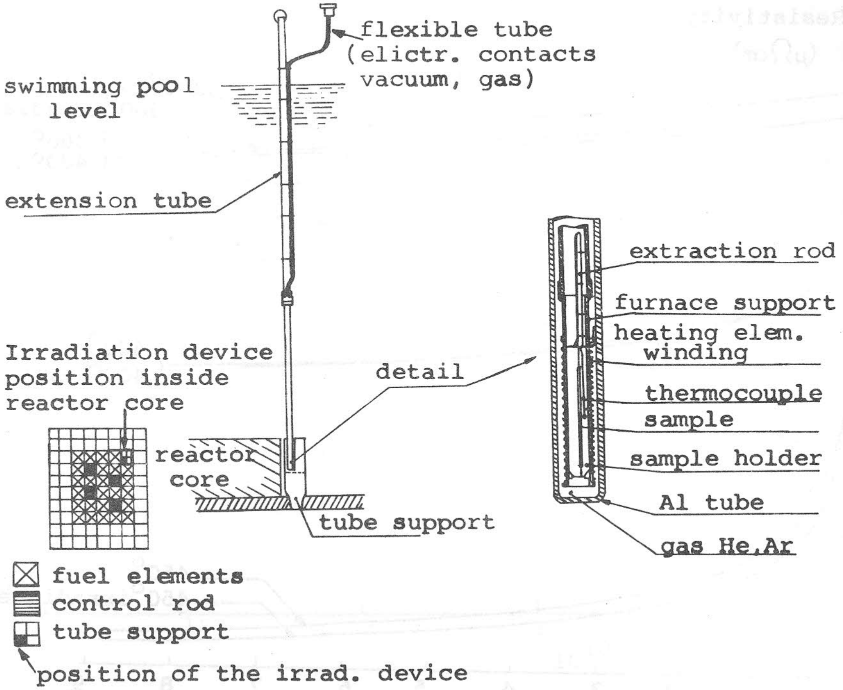

Fig.3 - Irradiation device for "in-situ" resistivity measurements.

The order formation on Cu Pd could only be detected by means of X - Ray diffraction. Computer analysis of the relaxation curves suggests existence of two predominant processes - nucleation and growth of ordered domains.

3.2 - Magnetic Methods.

With the aim of establishing characterization methods of nuclear materials, basic research theory and techniques (solid state physics) and applied techniques (irradiation and mettalurgy) were used to find some practical proceedures. This is the case of supersaturation curves, determined by means of the Magnetic After Effect (MAE), which makes feasible a cheap and rapid pre-selection of more adequate materials before the destructive stage of void formation and swelling. Since vacancy supersaturation is a necessary condition for the formation of voids, the metal or alloy with the lowest supersaturation is initially a more suitable material nuclear envi

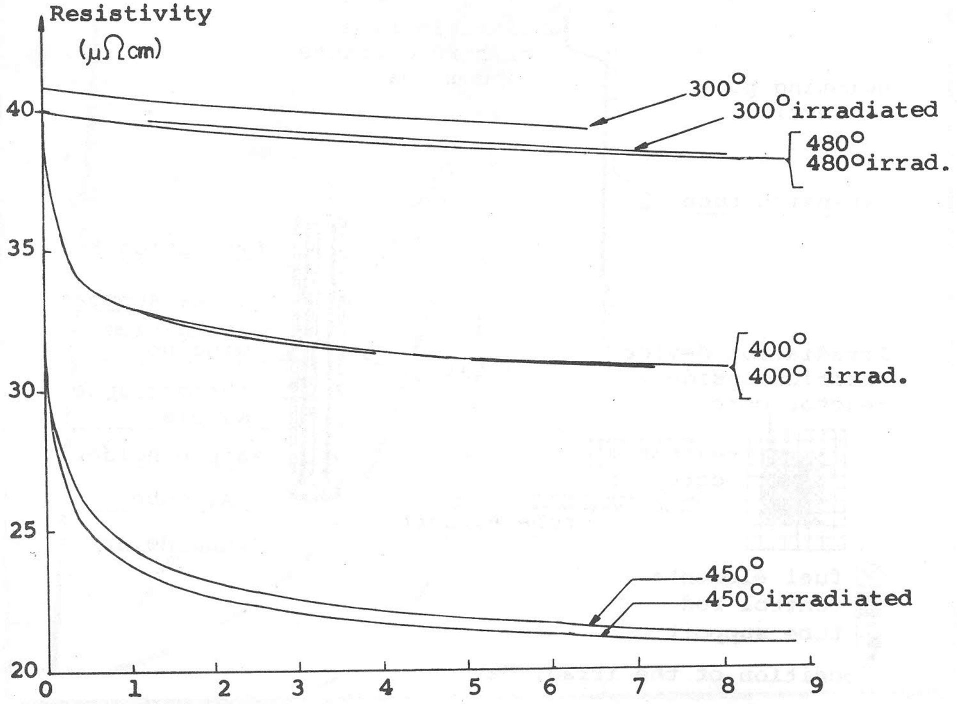

Fig. 4. - CuPd ordering with and without irradiation

ronments.

The relaxation time constants τ are inversely proportional to the vacancy concentration C_v,

$$\frac{1}{\tau} \propto C_v \qquad (4)$$

The supersaturation is defined as the ratio between the vacancy concentration under irradiation $C_{v\ irrad.}$ and the thermal vacancy concentration C_v,

$$S = \frac{\tau}{\tau_{irrad}} = \frac{C_{virrad}}{C_v} \qquad (5)$$

giving on Fig.6 the supersaturation curves for FeNi (50-50 at%), FeNi Mo (50-50 at%+50 ppm) and FeNi Cr (49,95-49,95 - 0,1 at.%).

An example of the improvement ("beneficial effect") (1) (2)

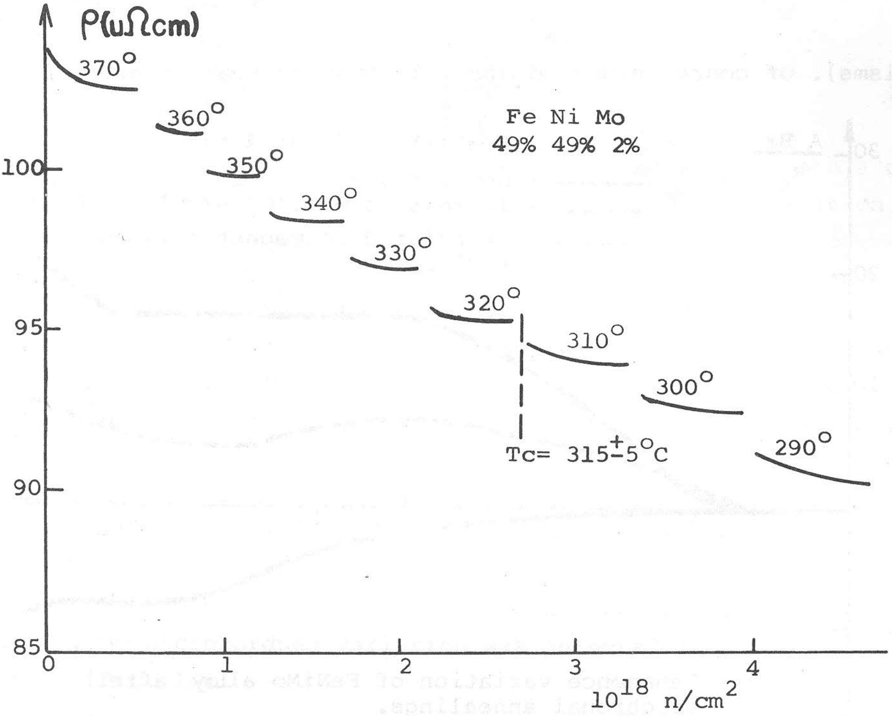

Fig.5 - Order-disorder transition T for Fe Ni Mo

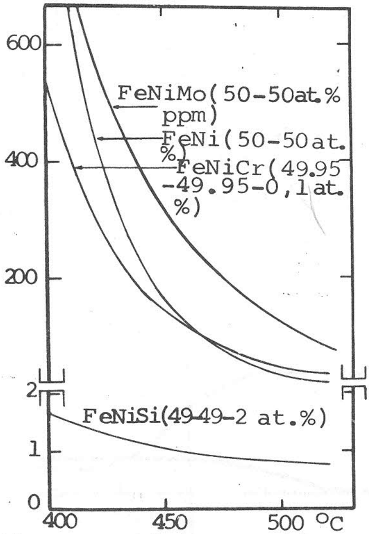

Fig.6. Vacancy supersaturation in FeNi alloys (59).

of the magnetic properties after irradiation Si and Mo doped FeNi alloy can be seen in Figs 7 and 8. In fact the small dose (4.10^{17} n/cm^2) fast neutron irradiation enhanced the short range ordering of the alloys increasing their ferromagnetic character and turning them more "trustful" for technological applications (e.g. - as servomech anisms). Of coure, higher doses will destroy that order.

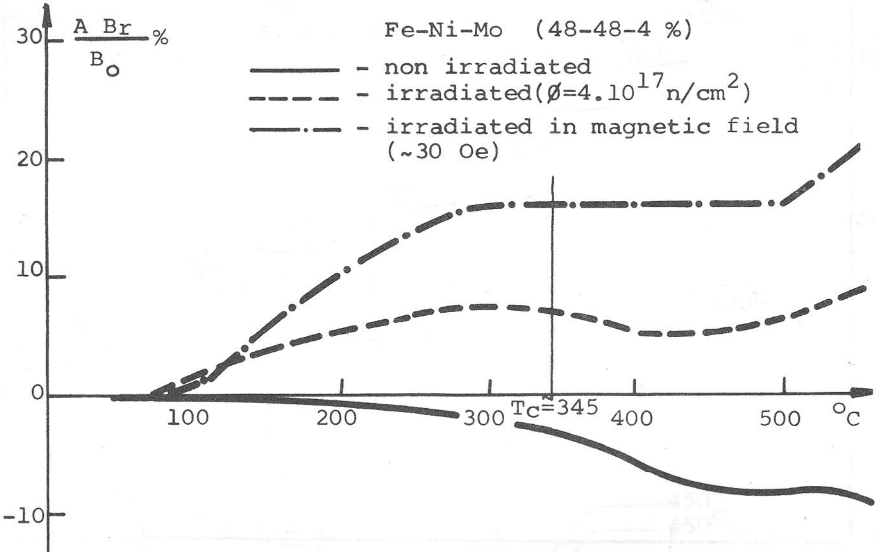

Fig. 7 - Remanence variation of FeNiMo alloy after isochronal annealings.

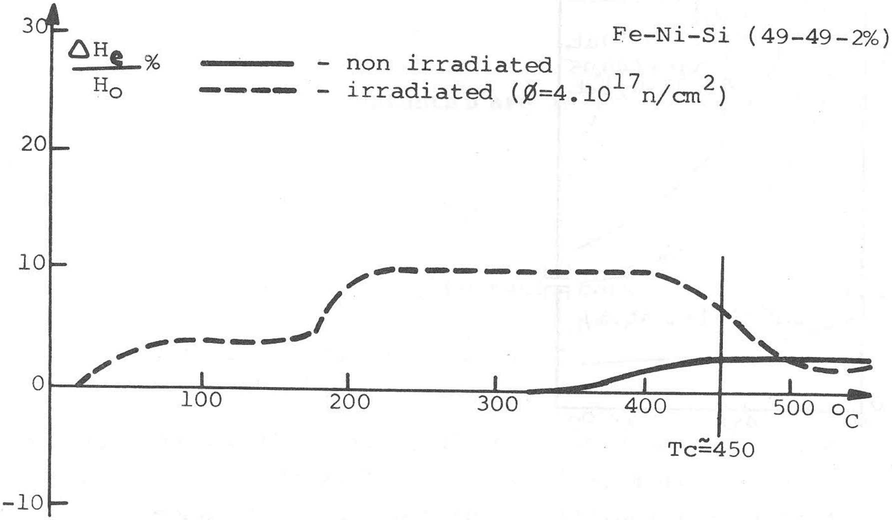

Fig.8- Coercitivity variation of FeNiSi, after isochronal annealings.

3.3 - Mechanical Methods

3.3.1 - Microhardness (46)(54)

The purpose of this experimental series was to make a contribuition to the technology of fabrication and characterization of special stainless steels with Niobium additions by means of microhardness (and electrical resistivity) studies-before, during and after - fast neutron irradiation. Effects of radiation in a wide range of temperature in austenitic stainles steel type AISI 321 with 0.05 and 0.10 wt.% Nb were investigated after neutron irradiation with fluences about 10^{17} n/cm^2 employing Vickers microhardness technique which is sensitive to microstructure changes produced by irradiation, as well as, thermal and mechanical treatments.

The results indicate the existence of radiation damage peaks around 480°C for the material without Nb additions; around 500°C with 0.05 wt.% Nb and around 570°C with 0.1 wt.% Nb, as shown in Fig. 9. No microhardnes variation was detected on non-irradiated material (Fig. 10)

The results were confirmed by means of electrical resistivity (Fig.11) The inversion of damage peaks is attribuited to the niobium carbide precipitates formed at grain boundaries. A displacement of the damage peaks to higher temperatures as a function of increased Nb addition was observed and can be considered as result of practical significance in nuclear materials selection.

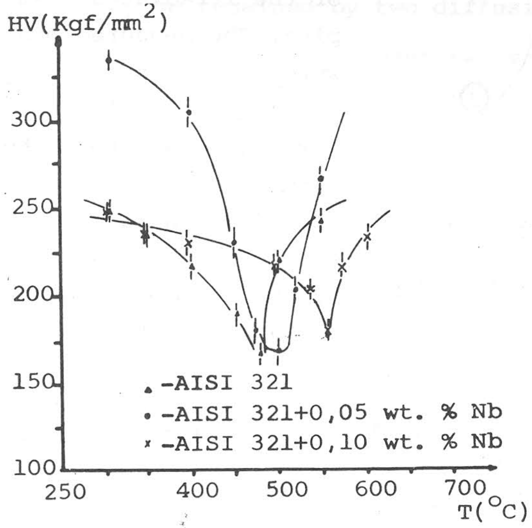

Fig.9- Variation of microhardness with irradiation temperature and composition.

3.3.2 - Ultrasound (65)(69)

The scope of using ultrasonic technique is to study the fast

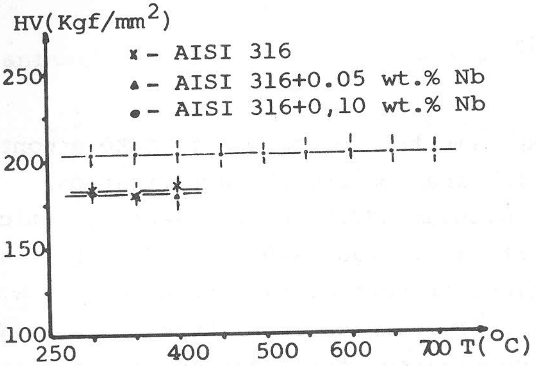

Fig.10- Microhardness VS. annealing temperature for the non- irradiated material.

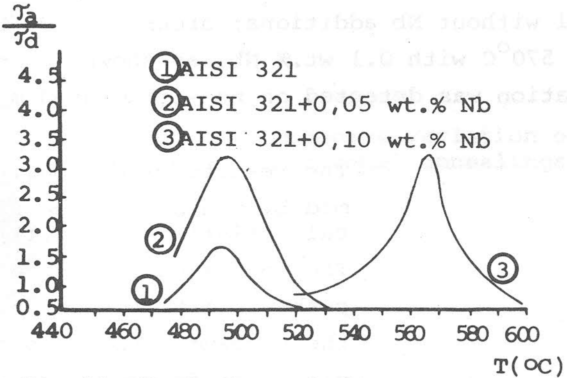

Fig.11- Variation of the ratio between the relaxation time constant after (τ_a) and during (τ_d) irradiation respectively, with temperature.

neutron radiation effects and temperature on Portlanf cement, trough the evaluation of the Dynamic Elasticity Module (Ed). Two methods were used:

a) Resonance Frequency and

b) Pulse Velocity

on non irradiated, as well as, samples irradiated to afluence of 7,2 10^{18} n/cm^2 (E ≅ 1 MeV), at a temperature of 120±5°C due to gamma heating. To eliminate the thermal effect this temperature was simulated in a microwave oven on the non irradiated samples, with the same number of thermal cycles of the irradiated samples. The measurements were performed in saturated state (relative humidity=100%) for the sake of comparison and showed a general decrease of Ed on both types of samples, allowing the distinction of the radiation effect in spite of the predominant thermal influence.

	Irradiated samples	non irradiated samples
Resonance Frequency Variation:		
Δ Ed (Fr)	- 24%	- 17%
Pulse Velocity Variation:		
Δ Ed (Vp)	- 28%	- 18%

Although from these data it not possible to describe the interaction of fast neutron irradiation with a complex structure like cement the radiation damage is evidenced by the smaller recovery of the dy namic elasticity module Ed, showing that the mechanical properties of cement are changed even by small irradiation doses.

3.4 - Simulation Experiments

The importance of simulation experiments has been described in in the introduction as an effective technique for materials characterization.

3,4.1 - Gas Release (53)(63)

Helium has been homogeneously introduced in Au, Ag and Al foil as room temperature by α- implantation in a CV-28 cyclotron to simulate (n,α) reaction. After implantation Helium release was observed during isothermal and linear heating experiments.

It is assumed that after room temperature implantation all He atoms are trapped in vacancies, i.e., in substitutional positions. Mobility may be regained by two diffusion mechanisms:

a) interstitial diffusion between two vacancies (substitutional positions) and,

b) vacancy mechanism assumed to operate in most substitutional alloys. For this case the activation energy ΔH_V is located in the following range (accordingly to the five frequencies model) (75).

$$Q_{2V}^{sd} - E_V^F \leq \Delta H_V \leq Q_V^{sd} \qquad (6)$$

whwre: Q_{2V}^{sd} - activation energy for self-diffusion by divacancies

E_V^F - formation energy of a vacancy

Q_V^{sd} - activation energy for self-diffusion

The activation anergy obtained through gas release clearly favours the vacancy macanism for single atom He diffusion as can be seen in Table 3.

3.4.2 - Stainless Steel Embrittlement by Alpha Particle Implantation in Cyclotron (66)(70).

Table 3. - Experimental activation energies (gas release) compared with calculated values for vacancy mechanism

metal	Vacancy mechanism		experimental values
	Q_{2V}^{SD} (73) $-E_V^F$ (74)	(eV) Q_V^{SD} (73)	H_{He} (eV)
Au	1,42	1,76	(1,70±0,13)
Ag	1,06	1,76	(1,51±0,12)
Al	0,92	1,28	(1,40±0,11)

The critical consequence of He gas, produced by (n,α) reaction, is the fast degeneration of the mechanical properties of metals and alloys. For this reason the study of this detrimental effect becomes important in:

a) selection of more adequate materials, through the characterizaton of presently available materials, and
a) search for new, more radiation-damage resistant alloys.

Two experimental series were performed in the Radiation Damage Division at IPEN-CNEN/SP on AISI 316 atinless steel, by means of creep measurements, in different conditions of; applied stress, temperature, He implanted doses and fast neutron irradiation. Creep $\dot{\epsilon}$ is a parameter strongly dependent on temparature and activation anergy E_a (of the order of self difusion), and is defined by:

$$\dot{\epsilon} \propto \exp \; (Ea \, / \, kT) \qquad (7)$$

The first experimental series consisted of ten samples, 100 µm thick, with 50% coldwork. Three samples (I-1, 2 and 5) were fast neutron irradiated inside the IEA-R1 reactor core with a fluence of $1{,}7.10^{18}$ n/cm^2; four samples (AI-1,2,3 and 4) were homogeneously He implanted at concentrations of 5 and 26 appm in CV-28 cyclotron and the rest (samples 20, 25 and 29) were control samples. The creep behaviour of the samples is visualized in Figs. 12 and 13.

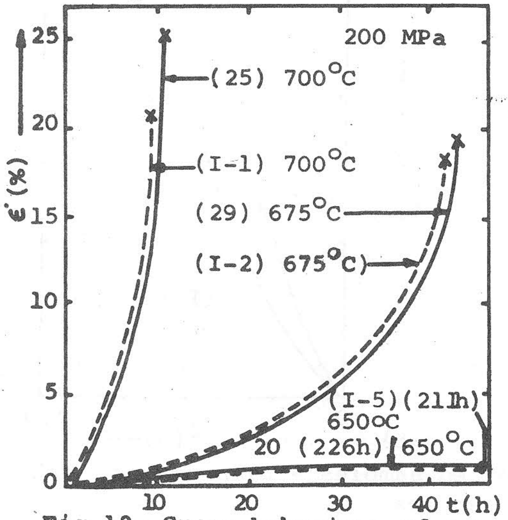

Fig.12- Creep behaviour of control samples (20,25 and 29) and fast neutron irradiated samples (I-1, 2 and 5)

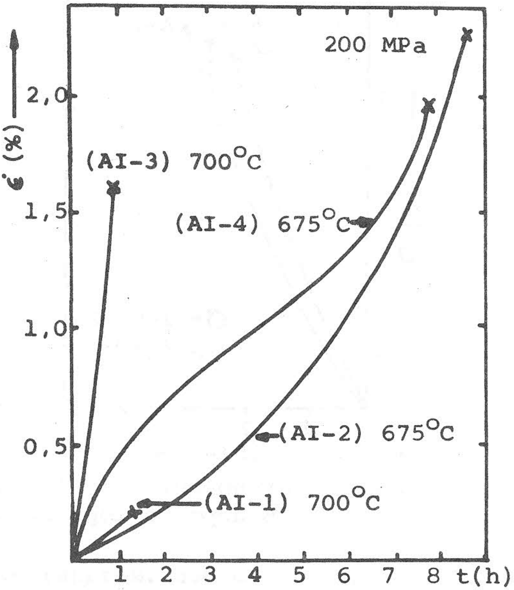

Fig.13- Creep curves on cyclotron implanted samples - with 5 appm (AI-3,4) and 26appm (AI-1,2) of He.

In Fig. 12, the neutron irradiated samples, except for the lower rupture point, have almost coincident creep behavior. On the other hand, in Fig. 13, a marked difference can be seen on He implanted samples by the decrease in rupture time and ductility, showing that embrittlement occurs even by small He concentrations.

The second experimental series consisted of ten samples with a 100 MPa applied stress, divided in two lots:

1 st. - 7 samples annealed during 2 hours at 1,050°C in vacuum batter than 10^{-5} Torr.

2 nd. - 3 samples 20% coldworked after the annealing

Two annealed (AI-1,2) and one coldworked (DI-3) samples were implanted with 26 appm He in cyclotron. The results of creep measurements can be seen in Figs. 14 and 15.

The analysis of the results ahows that:

- approximately, a factor 2 deformation (to rupture) decrease, and a factor 10 increase in rupture time on 20% coldworked samples (D) in comparison do the annealed samples (A).
- the implantation of 26 appm of He (samples AI-1,2 and DI-3) has a great effect on the reduction of rupture time (t_r), if compared with the coldworked samples (D-2,3,4); approximately a factor 4 t_r decrease

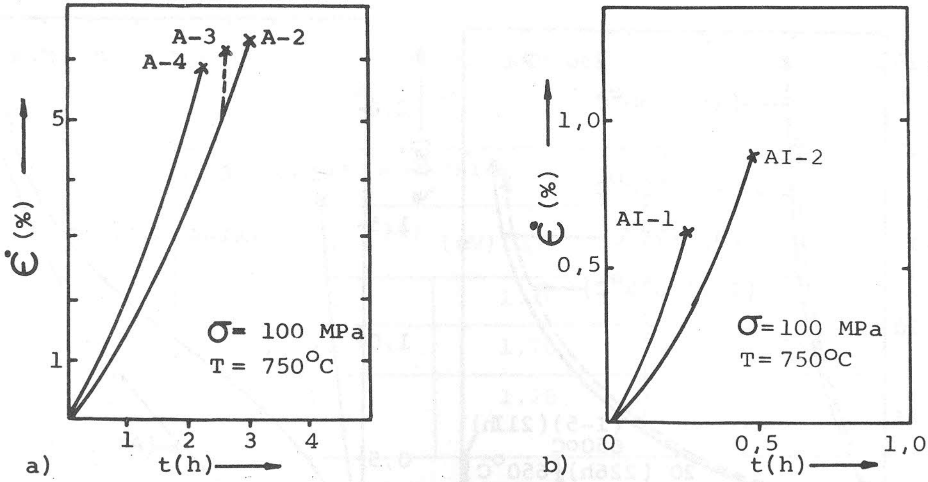

Fig.14- Creep curves: a) annealed samples - b) annealed and 26 appm He implanted samples

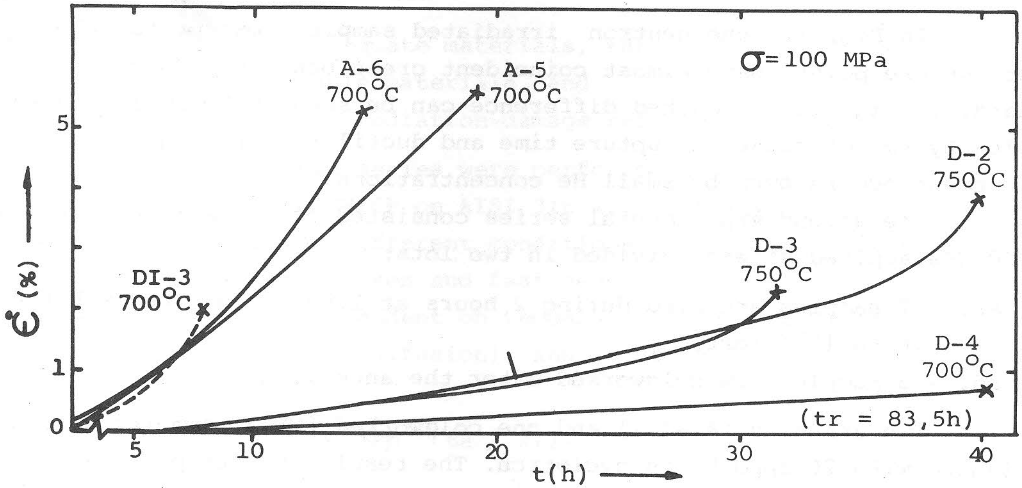

Fig.15- Creep behaviour on samples: annealed (A-5,6)- coldworked (D-2,3,4)- coldworked and 26 appm He implanted (DI-3)

for (AI-1,2) and a factor 2 tr decrease for (DI-3).

All the above mentioned results offer an experimental evidence of the intense embrittlement due to the presence of He in the materi als structure.

4. - OTHER RADIATION DAMAGE GROUPS IN BRASIL

The presentation of the work "Order-Disorder Transition in Cu Pd"(12) in 1966, initiated the studies of radiation damage of materials at IPEN in 1966 by simulating their performance under power reactor working conditions, i.e. during fast neutron irradiation ("in situ" experiments) at high temperatures (up to 700°C) and controlled atmosphere, and charecterizing their physical properties by means of electrical, magnetic, mechanical and microscopic methods.

The pioneering work on radiation damage after proton irradiation in cyclotron was performed by Suszczynski and Quaranta Cabral in 1974 (76). The merit of this experiment was to demonstrate:

a) the existence of high temperature (approx. 500°C) proton irradiation (E= 5MeV) hardening andconsequently embrittlement of the stainles steel 316 studied.

b) the possibility to simulate high fluences (long time in-pile irradiations) in only some hours (1 to 5 h) of proton irradiation in a cyclotron. In this experiment the proton fluence was 3.10^{18} p/cm^2.

At the Instituto de Física - from the Universidade Federal do Rio Grande do Sul - the research group headed by Prof. Zawislak is performing, among others, very interesting experiments on N_2^+ particles and α-implantation in pure iron (77). Fe samples were N_2^+ implanted at 400 KV ion implanter with different energies to allow a homogeneous implantation to a depth of 1000 Å and a concentration of approximately 40 at. %. The post-implantation of α-particles was made to a concentration of 1 at. %. The ion current, in all cases, was below 1 μA and temperatures were kept below 70°C. It was found that the post implantation of α- particles significantly alters the diffusion of nitrogen, depending on the type of nitride precipitates present in the structure. Since the Helium-Vacancy (He - V) clusters diffuse slowly below 600°C, the mobility of nitrogen is reduced by trapping at the He - complexes. This trapping effect is of potential application in metallurgy, where the retention of nitrides is of major importance, as well as, in nuclear technology due to a probable inhibition in the void formation processes.

At the cyclotron of the Instituto de Engenharia Nuclear - CNEN - Rio de Janeiro, a radiation damage group has formed under the leadership of Dr. Carneiro Gonçalves using the technique of positron anihilation. A time spectrometer has been installed to measure positron life-time in materials, as proposed technique of radiation damage study. Using a simulataneous gamma-ray source ^{60}Co, the resolution function with 390 ps FWHM and 1172 ps FWHM was obtained. Life-time measurements were performed in organic and inorganic materials, such as, cicle-hexan, teflon, sodium, steel, zinc, copper, aluminium and others. The results compared with those from the literature show that the time spectrometer is ready to be used in de proposed studies.(75)

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