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**Georgi Lucki e Valdir Sciani**

**DEPARTAMENTO DE FÍSICA E QUÍMICA NUCLEARES**

**CNEN/SP  
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# RADIATION EFFECTS ON METALS, ALLOYS, AND CEMENT<sup>(\*)</sup>

G.Lucki e V. Sciani

## ABSTRACT

High - energy particle irradiation of materials brings as a consequence changes in their atomic structures that alter the electrical, magnetic and mechanical properties which are the most important characteristics for practical applications of metals and alloys. A review is made on experimental results of in-pile (IEA-RI reactor) and CV-28 cyclotron irradiated materials. Resistivity measurements on CuPd and FeNi alloys showed different behaviour during fast neutron irradiation. While CuPd had almost coincidental relaxation curves, FeNi presented a distinguishable short and long-range ordering with the critical order-disorder temperature at 515°C. Vacancy supersaturation curves of FeNiSi (49-49-2 at.%), FeNiCr (49-95-49,95-0,1 at.%), FeNiMo (50-50 at.% + 50 ppm) and pure FeNi (50-50 at.%) , determined by means of the Magnetic After Effect are presented as an effective pre-selection method of nuclear materials before the destructive stage of void formation and swelling. A displacement of damage peak from 480 to 500 and 570°C was detected on pure AISI 321 stainless steel and with 0,05 wt.% and 0,10 wt.% of Nb additions by means of resistivity and micro-hardness. Ultrasound techniques applied to fast neutron irradiated portland cement paste (fluence  $7,2 \times 10^{18}$  n/cm<sup>2</sup>) showed a 24% decrease in its dynamic elasticity modulus. Helium diffusion on Au, Ag and Al foils irradiated in cyclotron was studied, suggesting a vacancy mechanism for single He atom diffusion. Embrittlement by Alpha particle implantation in cyclotron - to simulate in-pile (n,  $\alpha$ ) reaction - was measured by high temperature creep on AISI 316 stainless steel.

## EFEITOS DA RADIAÇÃO EM METAIS, LIGAS E CIMENTO

### RESUMO

A irradiação de materiais com partículas de alta energia tem como consequência mudanças na sua estrutura

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(\*) Trabalho apresentado no 4<sup>th</sup>. International Symposium on Radiation Physics , em São Paulo, de 03 a 07 de Outubro de 1988.

atômica o que altera as suas propriedades elétricas, magnéticas e mecânicas; as quais formam o conjunto das características mais importantes para as aplicações práticas de metais e ligas. No trabalho é feita uma revisão dos resultados experimentais de materiais irradiados no caroço do reator IEA-R 1 e no ciclotron CV-28. Medições da resistividade em CuPd e FeNi evidenciam comportamentos diferentes durante a irradiação com nêutrons rápidos. As curvas de CuPd eram praticamente coincidentes, enquanto as isotermas de FeNi mostram a ordenação de curto e longo alcance, com a temperatura crítica da transição ordem-desordem a 515°C. Curvas de supersaturação lacunar de FeNiSi (49-49-2% at.), FeNiCr (49,95-49,95 - 0,1% at.), FeNiMo (50-50% at.+50 ppm) e FeNi (50-50% at.) determinadas por meio de EFEITO MAGNÉTICO PROTÉRIO são apresentadas como um método efetivo de prevenção de materiais nucleares antes do estágio destrutivo da formação de cavidades e do inchaço. Deslocamento de picos de dano, de 480 para 500 e 570°C foi detectado no aço inox AISI 321 puro e com adições de 0,05% e 0,10% em peso de Nb, por meio da resistividade e microdureza. Técnicas ultrassônicas aplicadas a pasta de cimento Portland irradiado (com fluxo integrado de  $7,2 \times 10^{18} \text{ n/cm}^2$ ) mostraram um decréscimo de 24% no módulo de elasticidade dinâmico. Difusão de He em chapas finas de Au, Ag e Al foi estudada, sugerindo um mecanismo lacunar para a difusão de átomos do gás. Experimentos de simulação para simular a reação  $(n, \alpha)$  em reatores foram realizados para estudar a fragilização do aço inoxidável AISI 316 implantado com partículas alfa no ciclotron, por meio de fluência em altas temperaturas.

## 1. INTRODUCTION

Reactor components are exposed to high fluences of energetic particles which produce changes in their atomic structure. These disturbances, consequently, influence the electrical, magnetic and mainly mechanical properties (e. g. hardness, strength, 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 <sup>(1)</sup> <sup>(2)</sup> in metals and alloys, most of them are destructive justifying the expression "Radiation Damage" for the influence of radiation on materials.

Considering the irreversible trend towards 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 can be made on the vulnerability of metals and other materials, by following example: the core components of a fast breeder reactor are exposed to a fast neutron flux of approximately  $10^{15}$  n/cm<sup>2</sup>. sec., which results in about 300 displacements per atom (dpa) after 10 years of operation, practically destroying 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 ( $\sim 3 \cdot 10^{-4}$  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 does not produce structural changes in metallic materials, other than heat. This is not the case of ionic conductors, semiconductors and insulators whose physical properties are strongly influenced by electronic excitation.

The radiation damage study consists of two major, not completely overlapping, fields: basic research on pure materials and technological studies (data collection) on commercial reactor materials. Research workers are interest

ed in basic properties of point defects and structure changes, performing experiments at low-temperature, low-dose irradiations using elaborate methods to determine the atomic behavior of interstitials and vacancies. Very pure metals and single crystals are used as samples with one kind of predominant 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. In the last decade a convergence of both fields has been noted and is due to great advances recently made in determining the structure and basic properties of point defects in pure metals with the research presently turning to defect agglomerates 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 theoretical 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 acquired 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 power reactors are 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 and 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 starting to analyse the technological and economic aspects of fusion reactors where material considerations are of paramount importance <sup>(3)</sup>. 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 and 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<sup>2</sup>. sec and temperatures around 300°C, the radiation damage normally is 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<sup>2</sup> (fluxes up to  $10^{11}$  n/cm<sup>2</sup>.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 reactor pressure vessels<sup>(7)</sup> as shown in Fig. 1.

The diminished toughness<sup>(8)</sup> 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

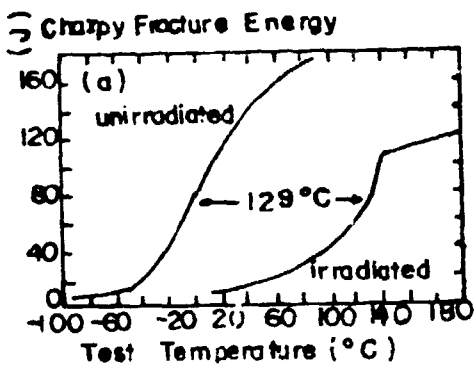


Fig.1-Ductile to brittle transition shift of Ni-Mo-Cr reactor pressure vessel steel after irradiation with  $1.25 \times 10^{20}$  n/cm<sup>2</sup> (7).

core and tested before the pressure vessel attains 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 interstitial atoms), however the role of impurity atoms and other microstructural defects in stabilizing and affecting these clusters in commercial alloys is far from being completely understood. 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 ( $\sim 950^\circ\text{C}$ ) 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<sup>2</sup> sec and high mechanical stresses ( ~100 MPa ), which lead to material deteriorations that limit the lifetime of many components below economically 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 decreasing order of deterioration: - swelling by void growth - irradiation creep - high temperature embrittlement and radiation enhanced diffusion. Investigations are being carried out, on world-wide scale, to develop radiation-resistant materials whose properties vary within certain limits established by engineering requirements, as shown in Table 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 surrounding the plasma are exposed to high temperatures, intensive mechanical stresses and fast neutron fluxes higher than  $10^{14}$  n/cm<sup>2</sup>, depending on the wall energy loading which varies from 1 MW/m<sup>2</sup> to 7 MW/m<sup>2</sup> for different fusion reactor designs. However, it is already known that the neutron energy spectrum will be considerably higher than in a fission reactor due to the 14MeV neutrons produced by fusion. These

will yield a strongly enhanced Helium production by (n,  $\alpha$ ) reaction leading, as a consequence, to high - temperature embrittlement.

Table I. Anticipated Structural Material Requirements for Future Reactor Systems (after Ref. (9)).

| PARAMETER   | UNIT               | FAST BREEDER<br>(steel) | FUSION REACTOR   |
|---|--------------------|-------------------------|--|
| Temperature                                       | $^{\circ}\text{C}$ | 300-600                 | 300-500(steel)<br>500-1000(refract)  |
| Maximum instantan displacement rate               | dpa/s              | $\sim 10^{-6}$          | $3 \times 10^{-7} - 10^{-4}$ (magnetically confined)<br>1-10 (inertially confined) |
| Avarange displacement rate                        | dpa/year           | $\sim 50$               | 10 - 30  |
| He production                                     | appm/year          | $\sim 10$               | 200-600(steel),<br>25-150(refract.)  |
| No. of power cycles                               | Year $^{-1}$       | $\sim 10$               | $10 - 10^5$ (magnetically confined) $10^7 - 10^9$ (inertially confined)            |
| Stress level                                      | MPa                | 60-120                  | 60-200   |
| Maximum permissible:<br>Volume change (life time) | %                  | <5                      | < 10   |
| Creep strain (life time)                          | %                  | <1                      | <1   |
| Ductility (elongation)                            | %                  | <1                      | <1-  |

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

Light ions, like p, d and  $\alpha$ , with energies from 5 to 30MeV are suitable for homogeneous damage production in 25 to 100  $\mu\text{m}$  thick samples appropriate 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<sup>(10)</sup>. With  $\alpha$  implantation the highest app He/dpa rates ( $\sim 10^4$  appmHe/dpa) can be achieved for simulation studies of He embrittlement in materials<sup>(11)</sup>. The high - voltage electron microscopy (HVEM) is especially useful as a simulation technique since it permits the observation of structure changes during the radiation damage generation with fast electrons ( $\sim 1\text{MeV}$ ). 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 implantations and the results obtained have shown that simulation techniques can provide fast, inexpensive and useful information about many aspects of radiation damage problem. Particularly, basic studies, as well as technological characterization for material development programs would be unthinkable without the use of simulation techniques.

Table II. Characteristic Features of Different Simulation Techniques (10).

| PARTICLE; EN<br>ERGY RANGE;                             | ADVANTAGES   | DISADVANTAGES   | MAIN FIELD<br>OF APPLICATION   |
|---|--|---|--|
| Heavy ions (se<br>lf ions); 2-5<br>MeV Van de<br>Graaf  | Extremely hi<br>gh damage ra<br>tes (100 dpa<br>in 3 hours);   | Very small<br>ranges (1 $\mu$ m) in<br>homogeneous<br>damage, surface<br>effects, un-<br>known distri-<br>bution of<br>high stresses  | TEM investi-<br>gation of<br>swelling by<br>void forma-<br>tion; screen-<br>ing of swell-<br>ing resistant<br>alloys   |
| Light ions (p,<br>d, $\alpha$ ) 5-30 MeV;<br>cyclotrons | Somewhat hi-<br>gher damage<br>rates than<br>reactors; ho-<br>mogeneous<br>damage in<br>~100 m thick<br>samples; me-<br>chanical tes-<br>ts possible | Limited to 5<br>dpa; damage<br>structure dif-<br>ferent from<br>n-damage  | Simulation of<br>"inpile" me-<br>chanical pro-<br>perties chan-<br>ges (irradi-<br>ation creep,<br>fatigue, em-<br>brittlement,<br>influence of<br>stress on<br>swelling). |
| Electrons;<br>0.2 - 3 MeV<br>HVEM                       | High damage<br>rates; produc-<br>tion and ob-<br>servation of<br>defect struc-<br>ture simul-<br>taneously   | Very thin lay-<br>ers (surface<br>effects) and<br>small areas<br>(stresses); da-<br>mage struc-<br>ture very dif-<br>ferent from<br>neutrons (sin-<br>gle Frenkel<br>pairs) | In-situ ob-<br>servation of<br>the build up<br>of damage<br>structure:<br>dislocation<br>loops, voids,<br>cluster com-<br>plexes, etc.                                     |

## 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  $T > T_d$  ( $T_d$  - threshold energy) creating a Frenkel pair (vacancy-interstitial)

- b) Transmutation nuclear reaction produce considerable concentrations of foreign atoms within the material, such as Helium gas due to  $(n, \alpha)$  reaction playing 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 transferring 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:

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

where:  $M$  - 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 (average number of atomic displacements produced per PKA with recoil energy

$T$  - is zero for  $T < T_d$ .)

$\phi$  - fluence (integrated flux)

$\sigma_d$  - displacement cross-section

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

$$C'_d = 1/\alpha_d \int_0^{T'} \int_0^{E_{max}} v(T) d\sigma(E,T) d\phi(E) \quad (3)$$

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

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

- ~ 50 eV for 1 MeV electrons
- ~ 5 KeV for 13 MeV  $\alpha$ -particles
- 50 KeV for 5 MeV Ni - ions
- ~100 KeV for 1 MeV neutrons

The physical meaning of these curves (Fig. 2) can be explained as follows: the steepness of the curves indicates the energy range of recoils with significant contribution to the damage: For example:

- 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.
- $\alpha$  - 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 departmental status - Materials Science and Technology Dept. In 1976 the Radiation Damage Division was created , performing the radiation Damage Studies on the compact cyclotron and reactor.

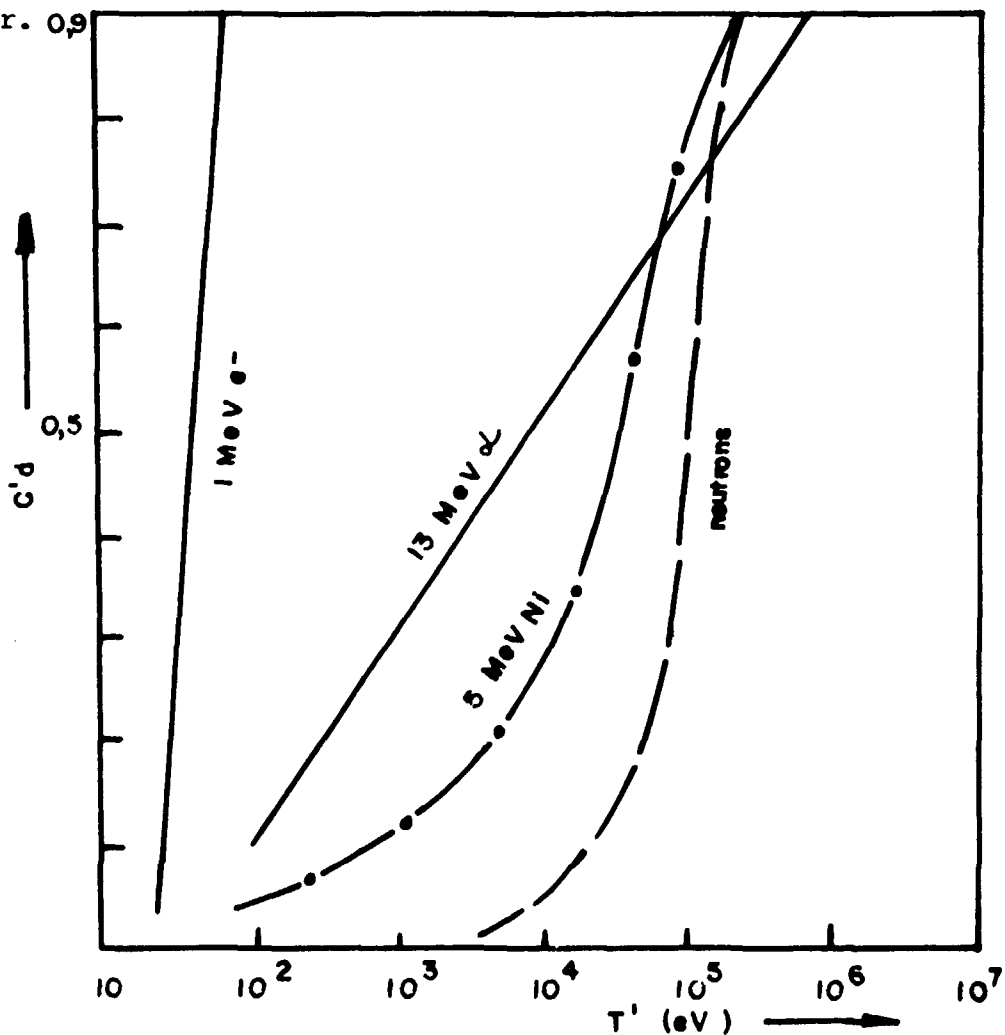


Fig.2. Function  $C'd$  calculated for Ni irradiated by different particles (10).

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 tempera-

tures 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 distinguishable short and long range order with the critical order-disorder transition at  $T_c = 515 \pm 5^\circ\text{C}$ .

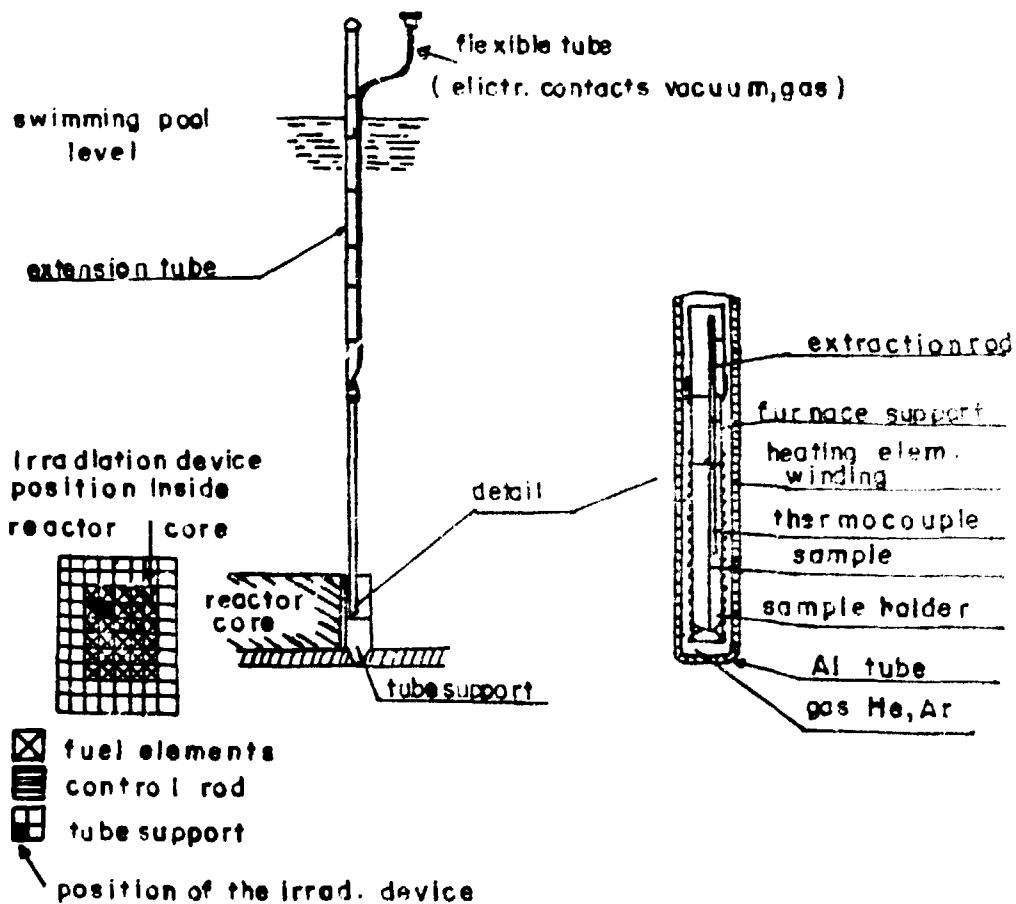


Fig.3. Irradiation device for "in-situ" resistivity measurements.

The order formation on Cu Pd could only be detected by means of X - Ray diffraction. Computer analysis of the relaxation curves suggests the 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 metallurgy) were used to find some practical procedures. 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 are attained. 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 for nuclear environments.

The relaxation time constants  $\tau$  are inversely proportional to the vacancy concentration  $C_v$ ,

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

The supersaturations is defined as the ratio between the vacancy concentration under irradiation  $C_{v \text{ irradiad}}$  and the thermal vacancy concentration  $C_v$ ,

$$S = \frac{\tau}{\tau_{\text{irradiad}}} = \frac{C_{v \text{ irradiad}}}{C_v} \quad (5)$$

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) of the magnetic properties after irradiation on Si and Mo doped FeNi alloy can be seen in Figs 7 and 8.

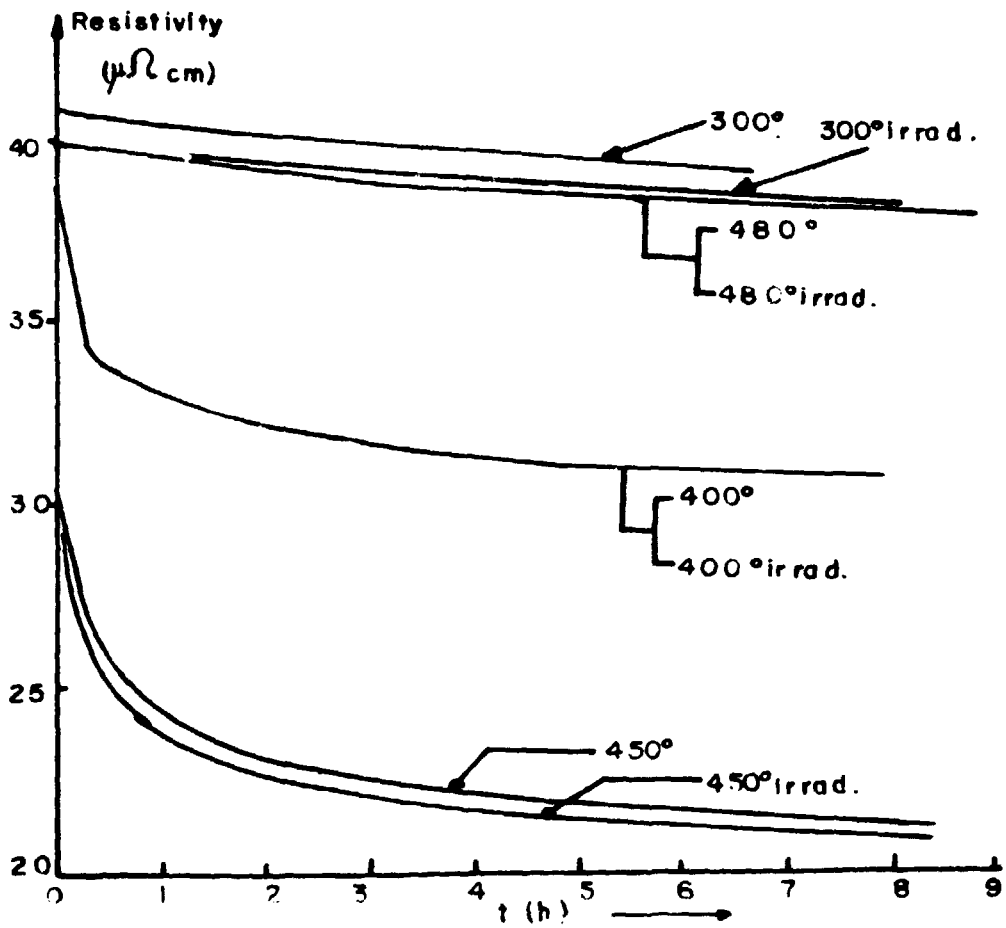


Fig.4. - CuPd ordering with and without irradiation.

In fact the small dose ( $4 \cdot 10^{17} \text{ n/cm}^2$ ) 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 servomechanisms ). Of course, higher doses will destroy that order.

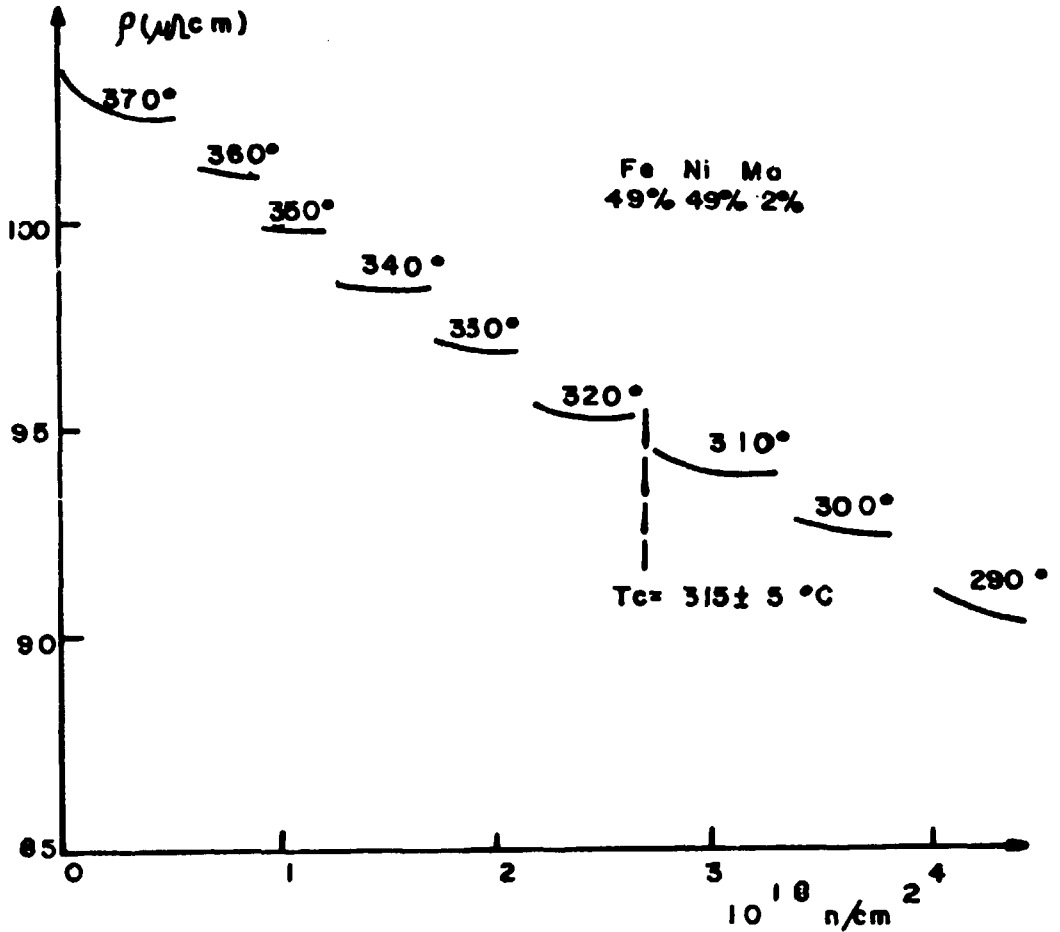


Fig.5 - Order-disorder transition T for Fe Ni Mo

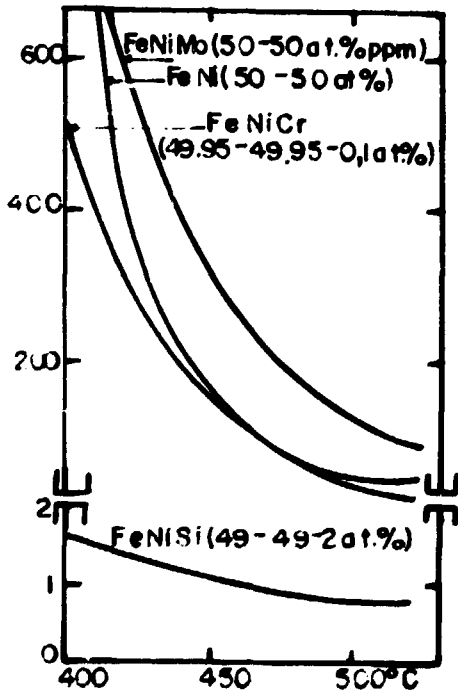


Fig.6. Vacancy supersaturation in FeNi alloys (59).

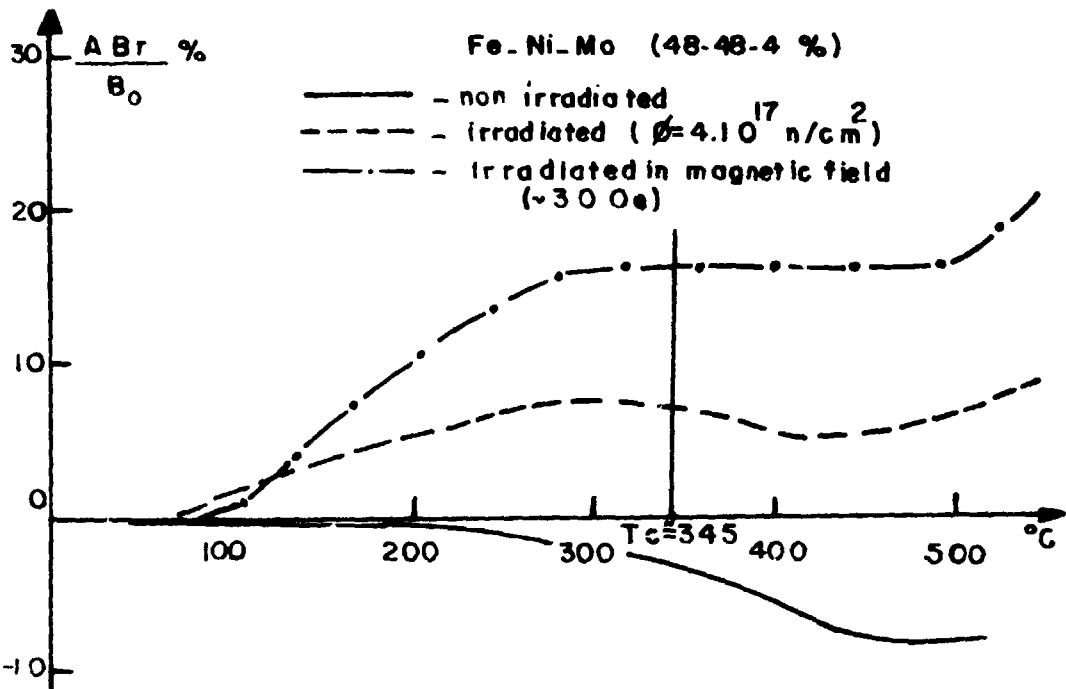


Fig. 7 - Remanence variation of FeNiMo alloy after isochronal annealings.

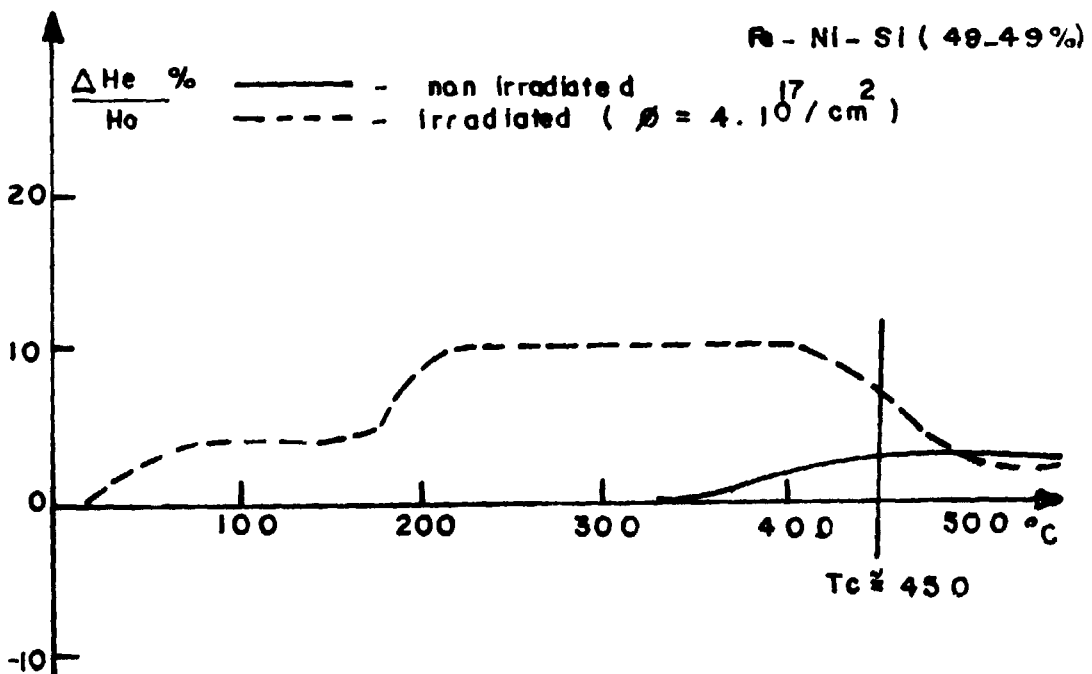


Fig. 8 - Coercitivity variation of FeNiSi, after isochronal annealings.

### 3.3 Mechanical Methods

#### 3.3.1 Microhardness (46) (54)

The purpose of this experimental series is to make a contribution 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 stainless steel type AISI 321 with 0.05 and 0.10 wt.% Nb were investigated after neutron irradiation with fluences about  $10^{17}$  n/cm<sup>2</sup> 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 microhardness variation was detected on non-irradiated material (Fig. 10)

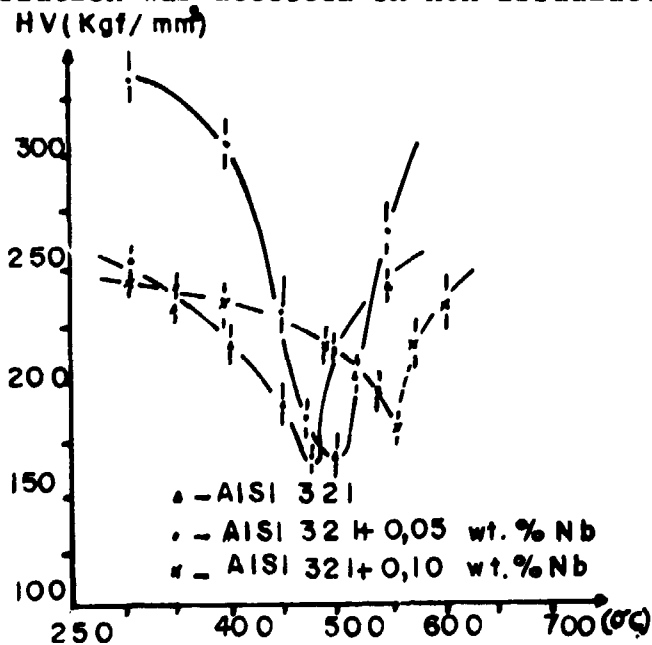


Fig.9-Variation of microhardness with irradiation temperature and composition.

The results were confirmed by means of electrical resistivity (Fig.11) The inversion of damage peaks is attributed 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.

3.3.2 Ultrasound (65) (69)

The scope of using ultrasonic technique is to study the fast neutron radiation effects and temperature on Portland cement, through the evaluation of the Dynamic Elasticity Module (Ed). Two methods were used:

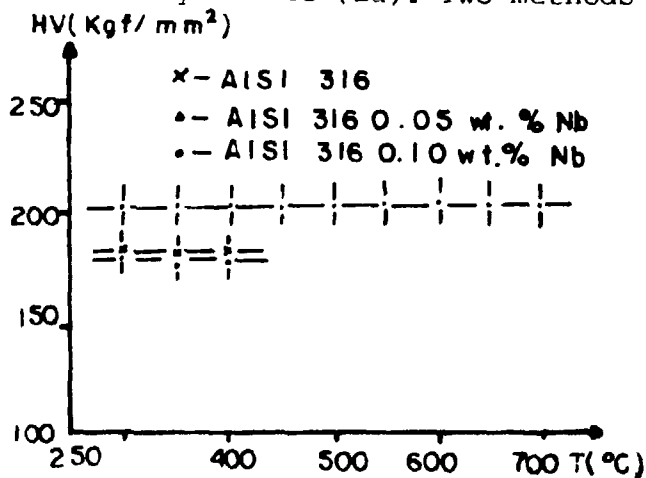


Fig.10- Microhardness vs. annealing temperature for the non-irradiated material.

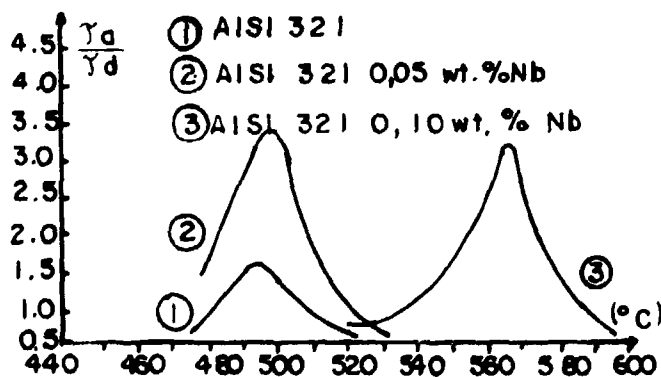


Fig.11- Variation of the ratio between the relaxation time constant after ( $\tau_a$ ) and during ( $\tau_d$ ) irradiation respectively, with temperature.

a) Resonance Frequency and

b) Pulse Velocity on non irradiated, as well as, samples irradiated to afluence of  $7,2 \cdot 10^{18} \text{ n/cm}^2$  ( $E \approx 1 \text{ MeV}$ ), at a temperature of  $120 \pm 5^\circ \text{C}$  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  $E_d$  on both types of samples, allowing the distinction of the radiation effect in spite of the predominant thermal influence.

|                                | <u>Irradiated</u><br><u>samples</u> | <u>non irradiated</u><br><u>samples</u> |
|--------------------------------|-------------------------------------|---|
| Resonance Frequency Variation: |                                     |   |
| $\Delta E_d(\text{Fr})$        | - 24%                               | - 17%                                   |
| Pulse Velocity Variation:      |                                     |   |
| $\Delta V_d (V_p)$             | - 28%                               | -18%                                    |

Although from these data it is 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 dynamic elasticity module  $E_d$ , 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 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  $\alpha$  implantation in a CV-28 cyclotron to simulate  $(n, \alpha)$  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  $H_V$  is located in the following range (accordingly to the five frequencies model) <sup>(75)</sup>.

$$Q_{2V}^{sd} - E_V^F \ll H_V \ll Q_V^{sd} \quad (6)$$

where:  $Q_{2V}^{sd}$  - 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 energy obtained through gas release clearly favours the vacancy mechanism for single atom He diffusion as can be seen in Table 3.

Table 3. - Experimental activation energies (gas release) compared with calculated values for vacancy mechanism. ( $Q_{2V}^{SD}$ ,  $Q_V^{SD(73)}$  and  $E_V^F(74)$ ).

| metal | vacancy mechanism          |                 | experimental values |
|-------|----------------------------|-----------------|---------------------|
|       | $Q_{2V}^{SD} - E_V^F$ (eV) | $Q_V^{SD}$ (eV) | $H_{He}$ (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)         |

### 3.4.2 Stainless Steel Embrittlement by Alpha Particle Implantation in Cyclotron <sup>(66)</sup> <sup>(70)</sup>.

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 characterization of presently available materials, and
- b) 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 stainless 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 temperature and activation energy  $E_a$  (of order of self-diffusion), and is defined by:

$$\dot{\epsilon} = \alpha \exp \left( -\frac{E_a}{kT} \right) \quad (7)$$

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  $1,7 \cdot 10^{18} \text{ n/cm}^2$ ; 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 samples is visualized in Figs. 12 and 13.

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 better than  $10^{-5}$  Torr.

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

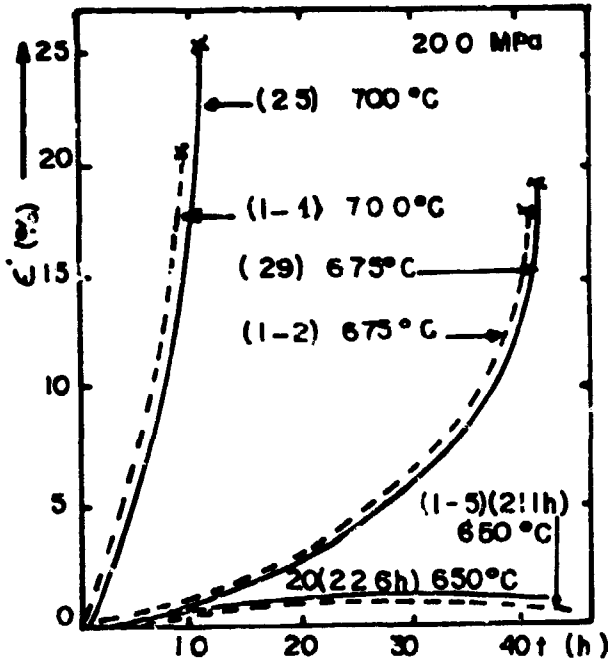


Fig.12-Creep behaviour of control samples (20,25 and 29) and fast neutron irradiated samples (I-1, 2 and 5)

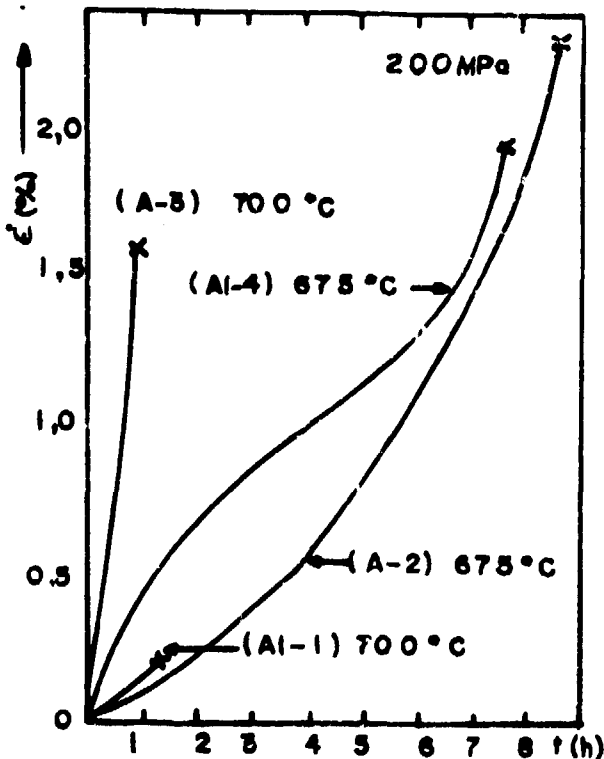


Fig.13-Creep curves on cyclotron implanted samples-with 5 appm (AI-3,4) and 26 appm(AI-1,2) of He.

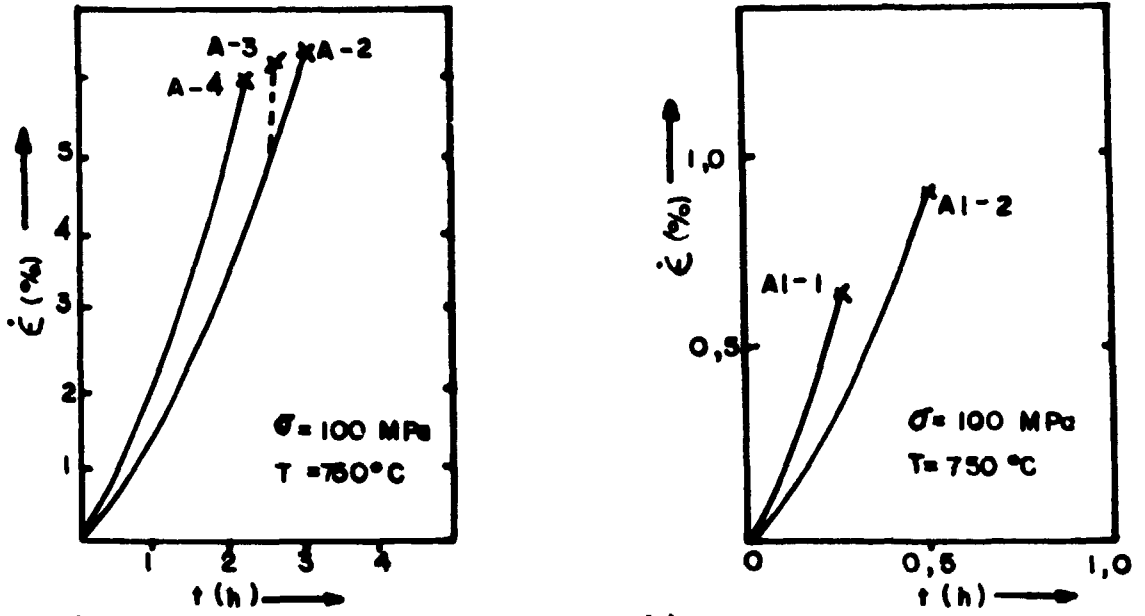
Two annealed (AI-1,2) and one cold-worked (DI-3) samples were implanted with 26 appm He in cyclotron. The results of creep measurements can be seen in Figs. 14 and 15.

The analysis of the results shows 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 for (AI-1,2) and a factor 2  $t_r$  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 mate-

rials structure.



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

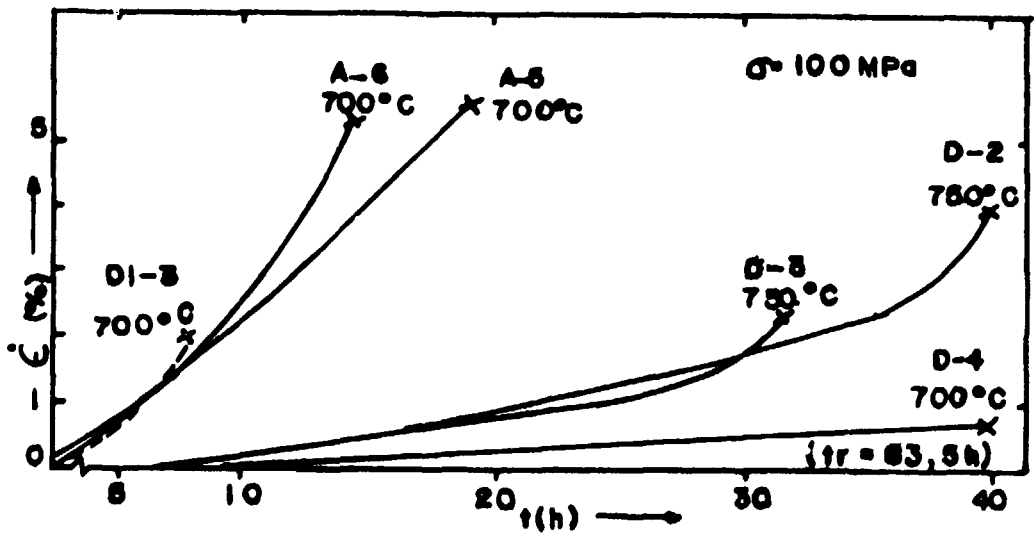


Fig.15- Creep behaviour on samples: annealed (A-5,6) - coldworked (D-2,3,4) - coldworked and 26 appm He implanted (DI-3).

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