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LA THERMOELECTRICITE ET L'EVALUATION DU VIEILLISSEMENT THERMIQUE DES ACIERS AUSTENOFERRITIQUES ET DU DOMMAGE D'IRRADIATION NEUTRONIQUE DANS LES ACIERS FERRITIQUES

APPLICATION OF THERMOELECTRICITY TO NDE OF THERMALLY AGED CAST DUPLEX STAINLESS STEELS AND NEUTRON IRRADIATED FERRITIC STEELS

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SYNTHÈSE :

Le pouvoir thermoélectrique (PTE) d'un alliage dépend principalement de sa température, de sa composition chimique et de sa structure atomique. La Direction des Etudes et Recherches d'Electricité de France, en collaboration avec l'Institut National des Sciences Appliquées (INSA), applique et améliore les techniques de mesure du PTE en vue d'étudier et de suivre deux phénomènes de dégradation affectant certains composants de la boucle primaire des réacteurs à eau pressurisée (REP).

Le premier de ces phénomènes est le vieillissement thermique des composants en acier inoxydable austénoferritique. La démixtion de la solution solide ferritique Fe-Cr-Ni est responsable de la diminution des caractéristiques mécaniques. Des études en laboratoire ont montré la sensibilité du PTE à ce phénomène métallurgique. Le PTE augmente de façon linéaire avec la teneur en ferrite et avec un paramètre de vieillissement de type Arrhenius, dépendant du temps, de la température et d'une énergie d'activation. Le PTE est également corrélé à certaines caractéristiques mécaniques telles la dureté et la résilience.

Le deuxième phénomène de dégradation étudié est le vieillissement des aciers ferritiques dû à l'irradiation neutronique à environ 290 °C. Dans ce cas, le mécanisme de dégradation est la formation de groupes d'atomes en solution et / ou de précipités riches en cuivre qui provoquent un durcissement du matériau. Une étude préliminaire des alliages binaires Fe-Cu irradiés par des électrons à 288 °C a révélé la possibilité de suivre l'appauvrissement en cuivre de la matrice ferritique. Il est en outre possible de suivre la restauration des propriétés mécaniques de l'alliage par recuit. Enfin, on a pu établir une corrélation entre la dureté Vickers et le PTE. Les mêmes tendances ont été observées sur les échantillons irradiés par neutrons du programme de surveillance de la centrale nucléaire de Chooz A.

EXECUTIVE SUMMARY :

The thermoelectric power (TEP) of an alloy depends mainly on its temperature, its chemical composition and its atomic arrangement. ~~The Research and Development division of Electricité de France, in collaboration with the Institute for Applied Sciences (INSA) uses and improves~~ The TEP measurement technique is used in order to study and follow two degradation phenomena affecting some components of the primary loop of Pressurized Water Reactors (PWR).

The first degradation phenomenon is the thermal aging of cast duplex stainless steel components. The demixing of the ferritic Fe-Cr-Ni slid solution is responsible for the decreasing of the mechanical characteristics. Laboratory studies have shown the sensitivity of TEP to the demixing phenomenon. TEP increases linearly with the ferrite content and with and Arrhenius-type aging parameter depending on time, temperature and activation energy. TEP is also correlated to mechanic characteristics.

The second degradation phenomenon is the aging of ferritic steels due to neutron irradiation at about 290 °C. In this case, the degradation mechanism is the formation of clusters of solute atoms and/or copper rich precipitates that causes the hardening of the material. As a first approach, a study of binary Fe-Cu alloys irradiated by electrons at 288 °C has revealed the possibility of following the copper depletion of the ferritic matrix. Moreover, the recovery of the mechanical properties of the alloy by annealing can be monitored. Finally, a correlation between Vickers hardness and TEP has been established. ~~The same tendencies have been observed for neutron irradiated samples of Chooz A's Nuclear Power Plant (NPP).~~

APPLICATION OF THERMOELECTRICITY TO NDE OF THERMALLY AGED CAST DUPLEX STAINLESS STEELS AND NEUTRON IRRADIATED FERRITIC STEELS

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Introduction

The degradation of the mechanical properties of metallic alloys during in-service aging is generally related to microstructural changes such as precipitation, apparition of crystal defects, etc ... These changes may have various origins: effect of temperature, effect of mechanical stresses, effect of irradiation, etc ... On the one hand, these mechanical changes may threaten the structural integrity of the component and so, may lead to its prematurate replacement, thus complicating the outage operations and increasing the maintenance costs. On the other hand, these mechanical changes may be lower than predicted so that the component lifetime is larger than expected. In the framework of lifetime extension and management, original non destructive methods are required in order to follow the aging kinetics of the component. For this purpose, indirect methods that consist in measuring the variations of a property sensitive to the aging phenomenon may be used such as, for example, the hardness or the resistivity measured by Eddy currents. However, the number of methods available for this testing are very limited and not always sensitive to the degradation mechanism.

The objective of our research work is to study the possibility of characterizing the aging kinetics by thermoelectric power measurements, and to develop a device performing these measurements on large components with enough accuracy as for the characterization of the materials evolution. In fact, the attempts to use such a method of investigation are very few (1-2).

After some general considerations on thermoelectricity, we mention the conventional method for TEP measurement on small samples and the principle of TEP measurement on large components. This last method has been improved through the development of a new non destructive device. It is original both in its conception and in its method of TEP calculation (3). Finally, we propose two examples of application of this device: the thermal aging of cast duplex stainless steels of the elbows of the primary loops of pressurized water reactors, and the effects of irradiation on the ferritic steels of reactor pressure vessels.

General considerations on Thermoelectricity

The thermoelectric power is a characteristic of the material that may schematically be described as its capacity to generate an electron flux when crossed by a thermal flux. This phenomenon is shown by Seebeck's effect. It appears in a circuit composed of two conducting materials A and B as represented in figure 1. When a thermal gradient is generated between the conductors so that the two junctions 1 and 2 are at the temperatures T_1 and T_2 respectively, and when the free ends of A are at the same temperature T_0, a measurable potential difference (V_2-V_1) appears between them. If T_2 is close to T_1, and if S_A and S_B are the absolute TEP of the materials A and B, the TEP of B with respect to that of A, at the mean temperature $T = (T_1+T_2)/2$, is given by the following relationship:

$$\Delta S = S_{BA} = S_B - S_A = (V_1 - V_2) / (T_2 - T_1) = \Delta V / \Delta T \quad (1)$$

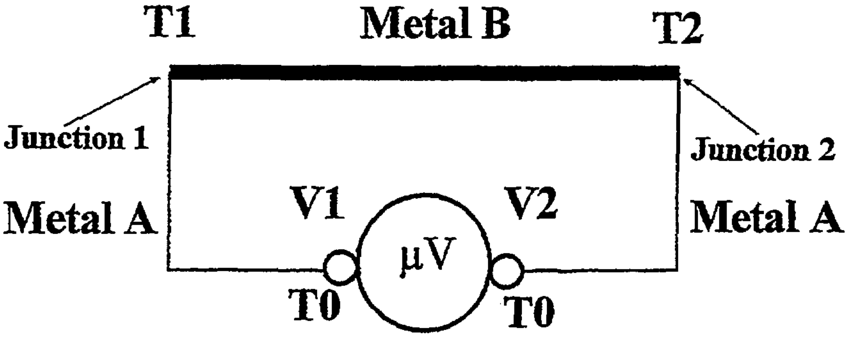

Figure 1. Seebeck's effect - Principle of TEP measurement

The absolute TEP of a pure metal after annealing may be positive or negative and may take values ranging from one μV/°K to a few dozens of μV/°K. Its variation as a function of the temperature is generally complex. In fact, two components with different origins contribute to the TEP: at low temperatures, a "phonon-drag" (or "network") component, related to the crystal network, that presents an extremum and, at higher temperatures, a diffusional component that often varies linearly with the temperature. All modifications of the crystal network of a metal may modify its thermoelectric power: change of crystallographic structure, introduction of impurity atoms, creation of dislocations, etc ... In the case of metal alloys, the thermoelectric power may be used as an investigation means in order to detect transformations even at a very small microstructural scale. The study of precipitation phenomena (4, 5), of solution treating, of short and long range ordering certainly represents one of the most interesting applications of thermoelectricity. The thermoelectric power may also be used to study the effects of cold working and of recovery during annealing or martensitic-type transformations.

These applications of thermoelectricity are achievable by using a TEP measuring system described in the next section (6). It requires to work on small samples. One advantage is the easiness and the rapidity of the measure which is moreover independent of the precise geometric shape of the sample.

Conventional TEP measuring device (small samples)

In order to measure the TEP of a conducting material, a system reproduces the diagram of Seebeck's effect (figure 1). A reference conductor, the TEP of which is known, (e.g. copper, aluminium) is taken as the conducting material A. The sample of unknown TEP is considered as the conducting material B. For this measuring device, the sample in the shape of a thin plate is set between two blocks of metal A maintained at temperatures T_1 and T_2 respectively (figure 2). The thermoelectric power is given by equation (1). This technique is called method « with lateral gradient ».

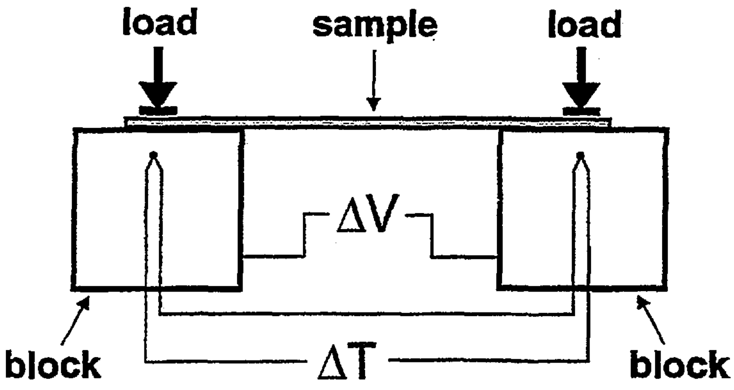

Figure 2. Lateral gradient method

The potential difference ΔV is measured between two wires of metal A connected to each block. The temperature difference ΔT is measured using two thermocouples inserted in each block at 1.5 mm under the contact surface with the sample. When taking blocks of a good thermal conducting material (e.g. copper, aluminium) and a sample with a small section with respect to its length, it is possible to reduce the thermal flux through the sample. So, the temperature difference measured by the thermocouples is only very slightly different from the temperature difference between the two contact points sample-block. The thermal gradient is homogeneously distributed along the sample between the contact points. The measurement gives the mean TEP of the part of the sample submitted to the thermal gradient.

The potential differences ΔV and ΔV_T (measured between the thermocouples) are amplified and used for the calculation of the TEP difference $\Delta S = S_B - S_A$ which is directly displayed by the device. The main difficulty is to measure a thermoelectric tension equals to a few µV thus requiring an amplifier with low noise and low thermal drift.

Such a device gives the TEP of the sample at 20°C with respect to a reference TEP with a good accuracy: the smallest detectable variation is about 0.002 µV/°K while the relative accuracy is 0.2%. It requires samples of small dimensions (typically 50 x 5 x 0.5 mm^3).

Local TEP measuring device (large components)

Principle

In order to develop a non destructive TEP measuring method, it is necessary to adapt the measurement principle described previously. Two test prods of a reference metal (e.g. copper), set at different temperatures, are applied on the surface of the component and generate a thermal gradient in the material (figure 3).

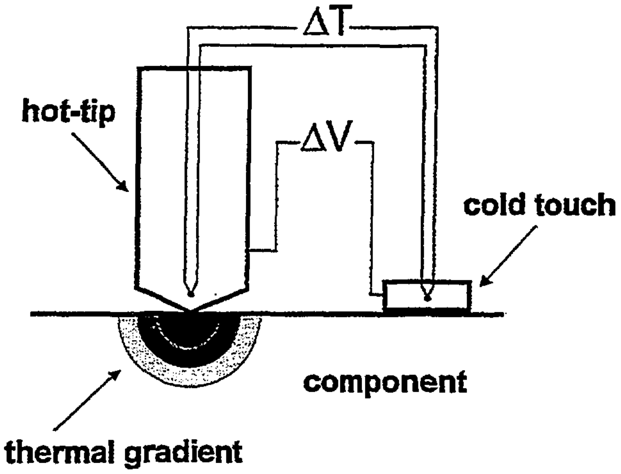

Figure 3. Hot tip method

One of the test prods has the shape of a tip (called « hot tip »). It is heated by a heating element connected to a temperature regulating system. The contact between the hot tip and the component is maintained with a given force. The contact area is spherical (radius r). The second test prod, set outside the heated zone, is simply pressed against the component and has approximately its temperature.

The step from the conventional method to the hot tip method complicates the precise determination of the temperature difference between the contacts test prod-component.

Method for TEP calculation

In order to determine the TEP of the heated portion of material, the temperature difference ΔT_M is measured with a thermocouple while the difference of potential ΔV_M due to Seebeck's effect is measured between two wires. The true TEP of the component with respect to that of the reference metal (test prods) at the mean temperature T is:

$$\mathrm{TEP}\ (T) = \Delta T_C \ / \ \Delta V_C, \tag{2}$$

where ΔT_C is the true temperature difference between the contacts « test prod-component » and ΔV_C is the thermoelectric tension that should be measured if the connecting wires had exactly the same TEP as the hot tip and the cold plate.

A thermal modelling allows to calculate the temprature distribution in the component and in the hot tip, and to determine a relationship between ΔT_C and ΔT_M. This correction depends on the tip geometry and on the thermal conductivity of the materials (component and test prods). Similarly, ΔV_C may be calculated from ΔV_M knowing ΔT_M et the precise TEP of the materials (wires and test prods).

In this model, the thermal resistance of the contact hot tip-component is neglected. This condition is obtained only for a correct choice of the surface state, of the axiality of the contact and of the applied force. For example, for a radius r = 0.3 mm, the value of the corresponding force is 30 N.

The choice of T_M results from a compromise between the obtention of a thermoelectric tension large enough and a low heating power. Moreover, it is more convenient to work in a temperature range where the TEP variations are linear as a function of temperature. Typically, a temperature variation of 25°C is suitable for a temperature of the component of 25°C.

A last correction is performed to obtain the TEP measurement at 20°C, so that it is independent of experimental conditions. For this, it is necessary to know the law of the TEP variations as a function of temperature. This law is linear in the considered temperature range. The TEP values obtained after correction may be directly compared to that given by the conventional TEP device. Moreover, the TEP value may be expressed with respect to a reference metal fully defined: for example pure copper annealed.

Area characterized by TEP measurement

The thermoelectric effect in the component is only obtained in the area concerned by the thermal gradient. When considering the area where 90% of the decrease of the temperature occurs, the characterized volume is a half-sphere with a radius R = 6 r. So, the choice of r may be achieved according to the volume to be characterized, the force being chosen accordingly. Small values of r may be chosen to study the heterogeneities of small dimensions and to realize a cartography. In order to perform a non destructive testing, we have chosen on the contrary the largest value of r (r = 0.3 mm) so that R = 1.8 mm.

Description of the hot tip device

As a first step, a benchmark system was performed in order to test the method for non destructive testing.

The hot tip and its heating element are fixed on a vertical carriage motorized which bring it in contact with the component while controlling the application force. The specimen to be tested is set on a copper plaque fixed on a X-Y motorized table. The cold touch is applied on one edge of the specimen. The measuring conditions are computed: up and down move of the hot tip, control of the applied force, positioning of the specimen, measurement of ΔV_M and ΔT_M, calculation of the TEP, measuring sequences, statistical data processing and cartography.

The basic components of this system (hot tip, applied force control system, cold plate and amplifiers) may be grouped together in a portable device for non destructive testing.

Conditions of use and results

The TEP measurement at a given location on the component surface is achieved as follows. The hot tip is first heated and regulated at a given temperature. It is brought into contact with the component and pressed until the chosen force is obtained (in about 20 s). Then, measurements of ΔV_M and ΔT_M are performed every second during 15 s. For each couple of values, the TEP is calculated at 20°C. Finally, the hot tip is moved upwards. The whole operation lasts about one minute.

In these conditions, we may consider that the measurements are performed during a quasi-standing regime as it was supposed for the thermal modelling. It means that the TEP does not change significantly when the measurements are performed beyond 15 s. However, the small total measuring time avoids a global heating of the component.

In order to reduce the random errors (nature of the contact hot tip-component) or errors related to defects in the homogeneity, a few measurements are performed at the same location and then at different locations on the component.

Validation tests have been achieved on three cylindrical components made of materials with different thermal conductivities:

- one 316L stainless steel, low thermal conductivity (16 $W.m^{-1}.K^{-1}$),
- one A60 carbon steel, medium thermal conductivity (49 $W.m^{-1}.K^{-1}$),
- one 2017 aluminium alloy, high thermal conductivity (167 $W.m^{-1}.K^{-1}$).

TEP measurements were performed on 50 points distributed all over the component diameter. The mean TEP (TEP_{HTM}) for each specimen is compared to that (TEP_{LGM}) obtained using the conventional device (lateral gradient method) in table 1.

The dispersion of the measurements is due on the one hand to the measuring uncertainties (0.06 µV/°K) and, on the other hand, to the inhomogeneity of the samples (in particular for the carbon steel). In the case of high thermal conductivity materials like the 2017 aluminium alloy, the measure is less accurate with a deviation of +4.6%.

Table 1: TEP measurements - Validation of hot tip method

Sample	**Conductivity** ($W.m^{-1}.K^{-1}$)	**TEP_{HTM}** (µV/°K)	**TEP_{LGM}** (µV/°K)
316L	16	- 2.66	- 2.68
A60	49	- 5.62	- 5.67
2017	167	- 3.41	- 3.26

The accuracy of our device gives the field of its application to non destructive testing taking into account the sensitivity of the TEP to the microstructural changes to be detected.

In order to insure a good reproducibility for the method, it is necessary to choose one or more reference alloys homogeneous and stable at room temperature. A stainless steel for which TEP measurements in different locations are not very scattered was chosen: a mean of five TEP measurements is enough to find the TEP value determined by the conventional system with an excellent accuracy. This reference specimen would allow us to correct any possible variation of the measurements after a very long time.

Applications

The TEP is strongly related to the aging of materials and thus, is directly correlated to the variations of the mechanical parameters characteristic of this aging. In the perspective of non destructive testing of large components, studies have been performed on steels using both devices previously described.

Thermal aging of cast duplex stainless steels of the primary loop of PWRs

Some cast components of the primary loop of PWRs are made of duplex stainless steel (austenite and ferrite). In-service temperature ranges from 285°C to 325°C. The chemical composition of this grade of steel is given in table 2. The ferrite content ranges from 5 to 25%. This alloy is characterized by a structure of solidification grains. Within each grain, colonies of austenite are surrounded by ferrite forming a continuous skeleton. The thermal aging in the temperature range between 300°C and 400°C affects the ferrite, where the unmixing of the ferritic Fe-Cr-Ni solid solution by spinodal decomposition and the precipitation of intermetallic G-phase particles rich in Ni and Si takes place (7). This aging causes an increasing of the hardness and a decreasing of the toughness.

Table 2: Chemical composition of cast duplex stainless steels (in weight percent)

Cr	**Ni**	**Si**	**Mn**	**C**	**Mo**
17 to 21	8 to 12	< 2	< 1.5	< 0.008	2 to 3

A study (8-10) of various products aged between 300°C and 400°C up to 30,000 hours has lead to the establishment of a linear relationship between the TEP on the one hand, and, on the other hand, the ferrite content δ and an Arrhenius-type aging parameter p depending on time t and absolute temperature T:

$$\mathrm{TEP}(\delta, t, T) = \alpha \,.\, \delta \,.\, p + \eta = \alpha \,.\, \delta \,.\, [\log_{10}(t) - Q/R.(1/T - 1/T_0)] + \eta, \qquad (3)$$

where α, Q, T_0 and η are constants calculated by best-fitting experimental data using least square method. R is the gas constant. Q represents an activation energy, T_0 a reference temperature and η the TEP of pure austenite ($\delta = 0\%$). Figure 4 shows the excellent correlation between the TEP calculated using equation (3) and TEP measured for 152 aged samples.

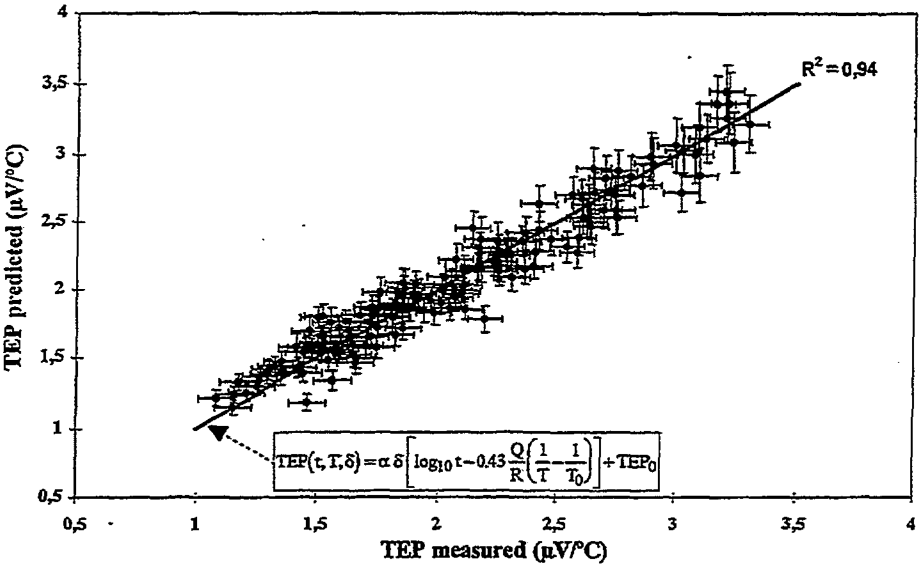

Figure 4. Correlation between TEP calculated using equation (3) and TEP measured

An example of aging evolution monitored by the TEP technique is shown in figure 5 for a product with a 28% ferrite content. The aging kinetics at 5 different temperatures ranging from 285°C to 400°C are represented.

The variation of the hardness ($Hv_{0,05}$) as a function of the TEP is represented in figure 6 for the same product as in figure 5 (ferrite content 28%). An excellent correlation is observed between the two parameters. Both hardness and TEP increase with aging time and temperature. The Hardness-PTE curve shows that TEP variations are large enough with respect to the variations of hardness to follow the aging of the component by non destructive testing.

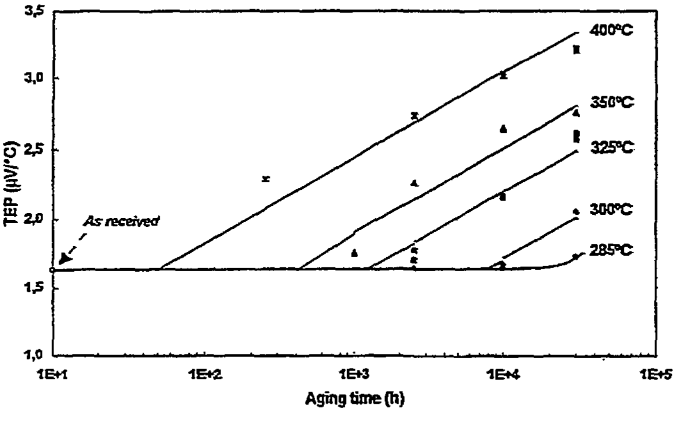

Figure 5. Thermal aging kinetics monitored through TEP measurements

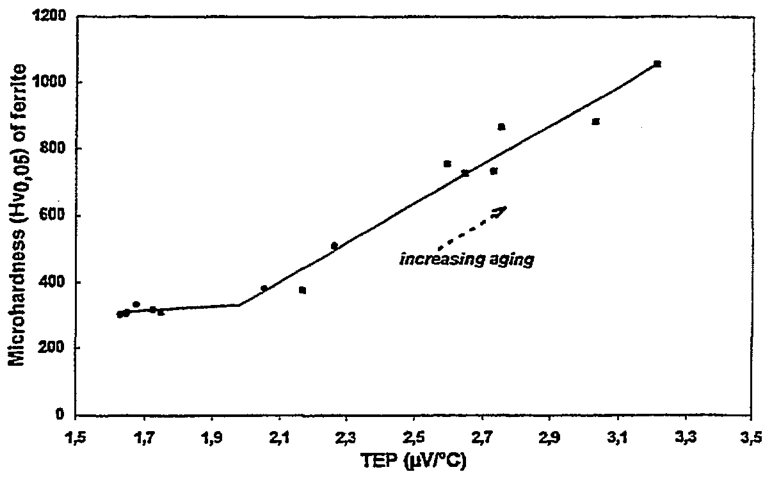

Figure 6. Correlation between Hardness and TEP (same product as in figure 5)

Aging of ferritic steels of RPVs by neutron irradiation

The reactor pressure vessel (RPV) of french PWRs is made of ferritic steel (AFNOR 16MND5). Under neutron irradiation, its mechanical characteristics change: the hardness, the yield stress, the tensile strength and the fragile-to-ductile transition temperature increase whereas the toughness decreases.

The irradiation favours the precipitation of some elements present in solid solution which should not precipitate at the considered temperature. A study (9,10) was achieved on a binary Fe-0.2%Cu alloy irradiated by electrons at 290°C. The copper is initially put into solution by holding the samples at 800°C and water quenching. For such a copper content, no precipitation due to thermal aging at 290°C is observed. However, during irradiation, an important hardening of the alloy, related to copper precipitation, is noticed as well as important TEP variations, related to the copper depletion of the ferritic matrix. Figure 7 shows the correlation between TEP and Hardness variations for the Fe-0.2%Cu alloy irradiated by electrons up to a fluence of 1.0 10^{20} e.cm^{-2}.

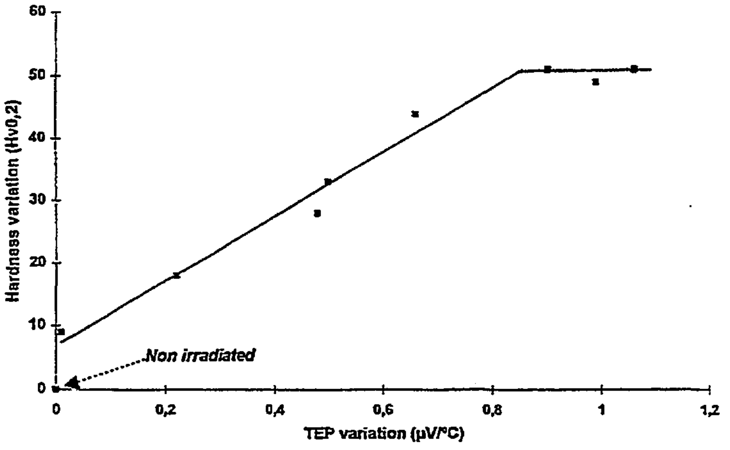

Figure 7. Correlation between TEP and Hardness variations
for a Fe-0.2%Cu binary alloy irradiated by electrons up to 1.0 10^{20} e.cm^{-2}

Following this study, the TEP measurement was applied to ferritic steels irradiated by neutrons. Five samples (fluence 0.0 to 15.0 10^{19} n.cm^{-2}) were taken from the survey program of Chooz A's Nuclear Power Plant (in-service temperature 265°C). A good correlation between the hardness and the TEP measurements is observed as shown in figure 8. For this diagram, the origin of both axis is the material in the non irradiated state. Both hardness and TEP increase with the fluence.

Four samples taken from the most irradiated state (15.0 10^{19} n.cm^{-2}) were annealed at 450°C for 0, 2, 20 and 100 hours respectively. During annealing, the copper atoms present in the clusters and/or copper-rich precipitates resulting from the irradiation are set back in solid solution, thus decreasing the TEP as well as the hardness (figure 8). After 100 hours at 450°C, the material almost recovers its mechanical properties.

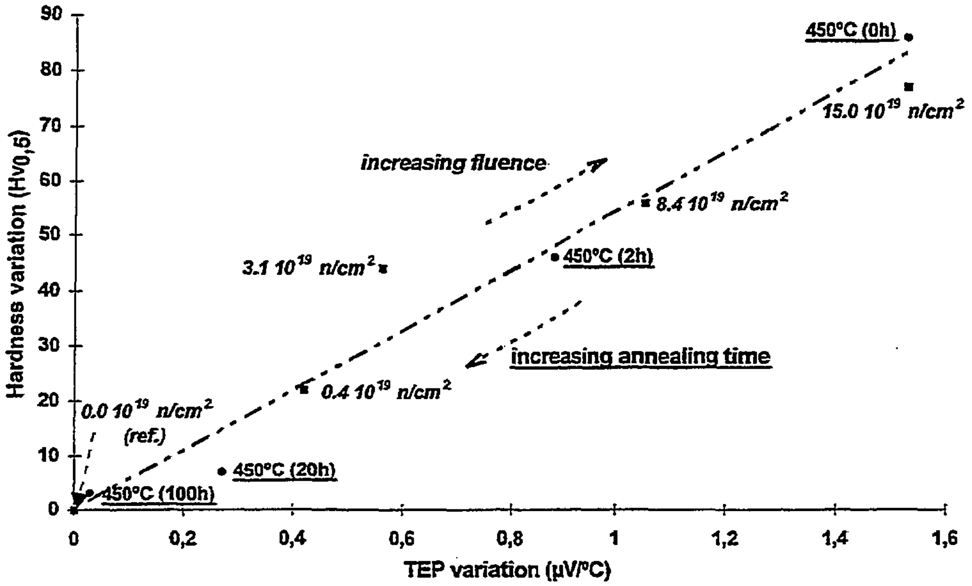

Figure 8. Correlation between Vickers Hardness and TEP for neutron irradiated ferritic steel of Chooz A. (■) symbols are for 0.0 10^{19} to 15.0 10^{19} n.cm^{-2} fluences and (●) symbols are for 0 to 100 hours at 450°C from the 15.0 10^{19} n.cm^{-2} irradiated state.

At least for Chooz A's ferritic steel, the TEP measurement is a good nondestructive means to follow the effects of neutron irradiation: the TEP increases with fluence. It may also be used to monitor the effects of annealing: the TEP decreases with annealing time and temperature.

The next step in the development of the TEP method is to extend the results obtained on Chooz A to other RPV steels. For this purpose, it will be necessary to study the effects of the microstructure and of the chemical composition of the steel on the TEP measurement and to enlarge data available on irradiated samples.

The TEP technique could be used as a nondestructive expertise means of irradiated materials. In fact, it would drastically decrease the important costs related to the manipulation and to the machining of irradiated samples.

Acknowledgement

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