Experimental Equipment for Studying the Residual Stresses Developed During High Temperature Reactions by X-Ray Diffraction

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Experimental Equipment for Studying the Residual Stresses Developed During High Temperature Reactions by X-Ray Diffraction

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Abstract

This paper describes a device dedicated to studying, by X-ray diffraction the residual stresses developed on surface samples as a function of temperature and atmosphere conditions.

The setup consists of :

a.) an horizontal axis goniometer which allows the programmed positionning of the sealed X-ray source and of the linear detector.

b.) a high temperature controlled atmosphere chamber.Particular attention has been paid to the thermal stability up to 1200°C and the accurate position on the sample.

INTRODUCTION

Residual stresses exist in all solids; They result from mechanical or chemical phenomena. At the moment, physico-chemical researchers are starting to pay attention to the part played, in surface reaction, by these stresses developed a few microns below the sample surface, in crystallised materials. X-ray diffraction is a privileged tool for investigating this domain. Measurements are carried out by using $\sin^2\psi$ technics [2,3].

Such determinations can be obtained as post-reactional events; i.e. the reaction being achieved is brought back to ambient temperature under standard atmosphere. This is a classical way to determine the residual stresses developed by thermal treatments or welding.

However, as residual stresses can result in cooling down, if one is dealing with the role of stresses on the reaction mechanisms, "in-situ" measurements are of basic interest.

According to the above point, we developed a high temperature, controlled atmosphere device specially built for residual stress investigations by X-ray diffraction. This apparatus was built at the laboratory "Réactivité des Solides" [1]. The basic goal for realising such a chamber was, first, to place the sample under thermal equilibrium with a heating spherical cavity of sufficient volume to ensure that the whole sample would not undergo a temperature - gradient greater than 1°C at 1000°C. Secondly, it was important for the positioning accuracy not to be modified by temperature dilatation of more than 0.1 mm between ambient temperature and 1000°C.

1.GENERALITIES

Determination technics are based on $\theta_{(hkl)}$ Bragg peak shiftings for macrostress determinations and on the peak broadening for microstress. These technics have been reviewed in [2,3]. From an experimental point of view, if one assumes that mechanics of continuous media can be applied at the lattice level and that the microcrystallographic constants of Young modulus and Poisson coefficient are available the stress field is deduced from the slope of the straight line $\Delta\theta$ versus $\sin^2\psi$. ψ is defined as the angle formed between the normal to the sample surface and the normal to the diffracting crystallographic planes.

2.APPARATUS

As represented in Figure 1, the apparatus consists of two parts :

- a horizontal axis goniometer with two motions by reference to the sample : $\theta_{(hkl)}$ Bragg angles and ψ angles and
- a motionless chamber with controlled temperature and atmosphere.

The guiding reasons are as follows :

- The horizontal axis cell allows an easy sample positioning. It is simply lying on a ceramic holder without any additional fastening.
- A motionless cell is required because a volume of more than 2 dm^3 is necessary to contain two heating elements of 500 Watts and the correlative cooling water jackets which are applied on the external walls of the chamber. In addition, that motionless chamber allows a direct connection with the turbo-molecular pump through a pipe 65 mm in diameter.

2.1 THE GONIOMETER

It is equipped with a standard sealed X-ray source and with an "INEL" linear detector whose window covers 12 ° on a focusing circle and is 400 mm in diameter. The X-ray source and detector are supported by two disks which can be moved, first, separately, to form a given Bragg angle θ and, secondly, to give, by reference to the normal of the motionless sample surface, simultaneously, a series of ψ angles from -45° up to +45°. These different movements are piloted through a computer by means of step-by -step motors.

Three different movements are described in the following figures (2a,2b,2c).

1.In goniometer initialisation, the X-ray tube and the detector are put in line on the horizontal plane.

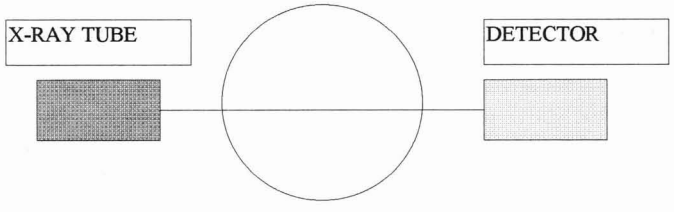

Figure 2a

2. The X-ray tube stays in its position whereas the detector moves to form the given Bragg angle 2θ. In motion Disk 2, which supports the detector, rotates using Disk 1 as a rack and pinion.

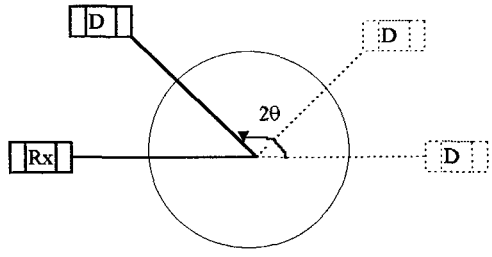

Figure 2b

3. There is a simultaneous motion of the X-ray tube (Disk 1) and of the detector (Disk 2) by rotating Disk 1 to give the series of programmed ψ angles (-45° to +45°).

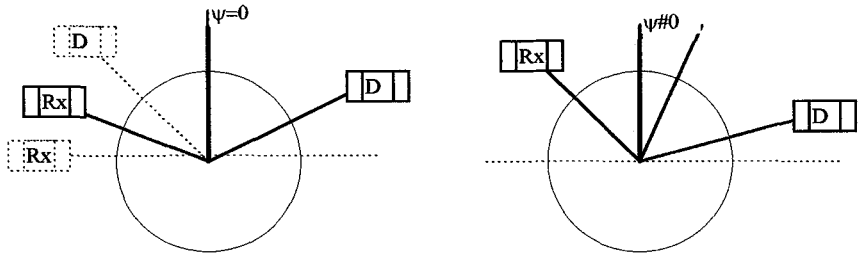

Figure 2c

Such a setting imposes several counterweights to balance the change in position of the X-ray tube and detector masses.

Two counterweights are used:

** *Counterweight n°1* balances the X-ray source. It consists of a mass placed at the end of an articulate arm, located under the stand. This arm is linked to Disk 1 by a cable wound on the disk. This device makes possible the X-ray source balance in any ψ positions (mass changes are in proportion to sinψ).

** *The counterweight n°2* is associated with Disk 2 and balances the detector mass. It consists of a string of identical mass hung on a cable which is wound on disk 2 (this system allows a variable balance depending on the position of Disk 2 : there is an unloading on the ground of some of the same masses). .

When the detector is in a horizontal position, the mass compensation by the counterweight is maximum.When the detector has gone past the vertical position, some balance is still applied in view of preventing a change in the points of contact between the disk and pinion teeth, as represented by Figures 3a and 3b schematically. If such a contact change should occur, as a consequence of the normal mechanical play, it would introduce an error in the measurements of approximately 10 channels on the linear detector. It represents 75 Mpa on an iron sample, measured with a chromium anticathode.

2.2 HIGH TEMPERATURE CHAMBER

The high temperature chamber is built in stainless steel with high vacuum technics. It consists of three parts : a central part (Figure 4) and two symmetric boxes (Figures 5 and 6).

** ***The central part*** supports the sample holder and two fixing pillars. It includes a 184° beryllium window for incident and diffracted X-ray beams. The sample holder of Figure 4 consists of :

a) a rotation modulus which allows horizontal ϕ angle rotations or oscillations of the sample.
b) a translation modulus, with a 0,01 mm accuracy, to position the sample surface in the goniometer axis.
c) a Pt/Pt 10% Rh thermocouple in contact with the sample.
d) a terminal alumina plate supporting the sample laid on a silica rod. The dimensions have been combined to compensate for dilatations.

** ***The two symetrical boxes*** (Figures 5-6) are cooled with water jackets. They contain a series of hemi-spherical screens and a central hemi-spherical ceramic cup fitted with a torus platinum heater. The maximum working temperature is 1200°C in an oxidising atmosphere or in a vacuum. In these conditions, the sample temperature homogeneity is greater than 1° at 1000°C.

A cathetometer is used for positioning the sample in the chamber centre with an accuracy of 0.01 mm.
The device is evacuated under a vacuum through a turbo-pump and a primary-pump (10^{-4}Pa).

3. STANDARDISATION

It consisted of verifying that the thermal expansion, of a given sample(i.e. copper), measured in the device is in agreement with the data published[4,5,6]. Such a result can be shown in Figure 7.

CONCLUSION

The device described above was successfully used to carry out « in situ » determinations of residual stresses at high temperature under oxidising atmosphere (1). In particular, it was possible to separate the part played by the cooling down from the reaction contribution in the residual stress field developed during high temperature oxidation of iron chromium alloys.

Acknowledgements

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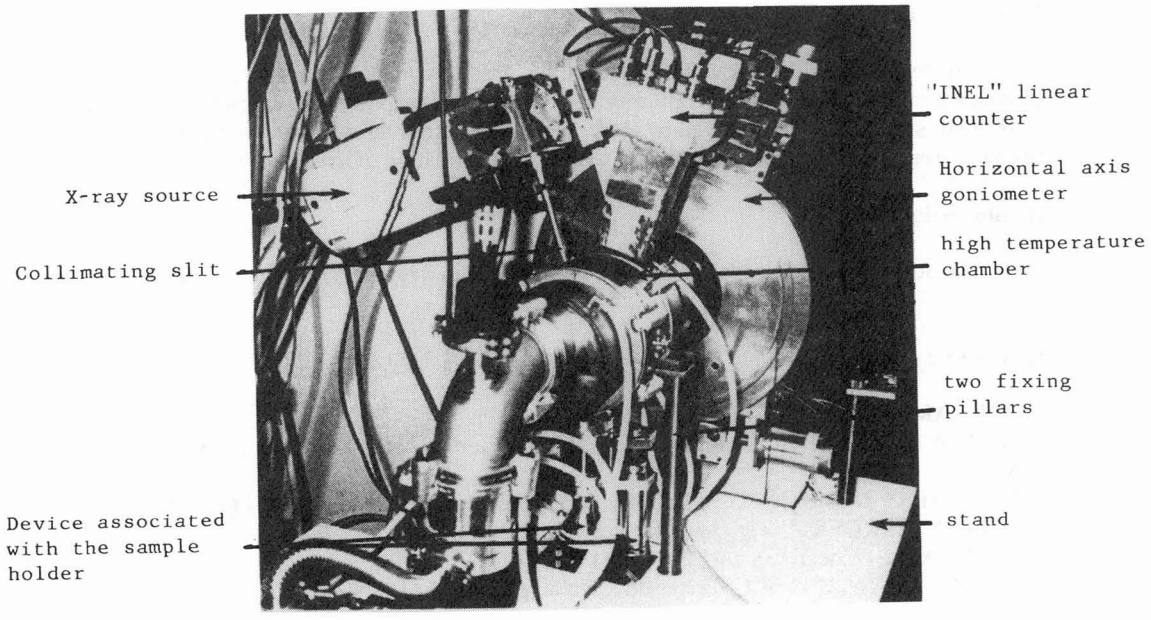

Figure 1 : General description of the goniometer θ/θ with a fixed sample on a horizontal axis, associated with a high temperature chamber working under controlled atmosphere.

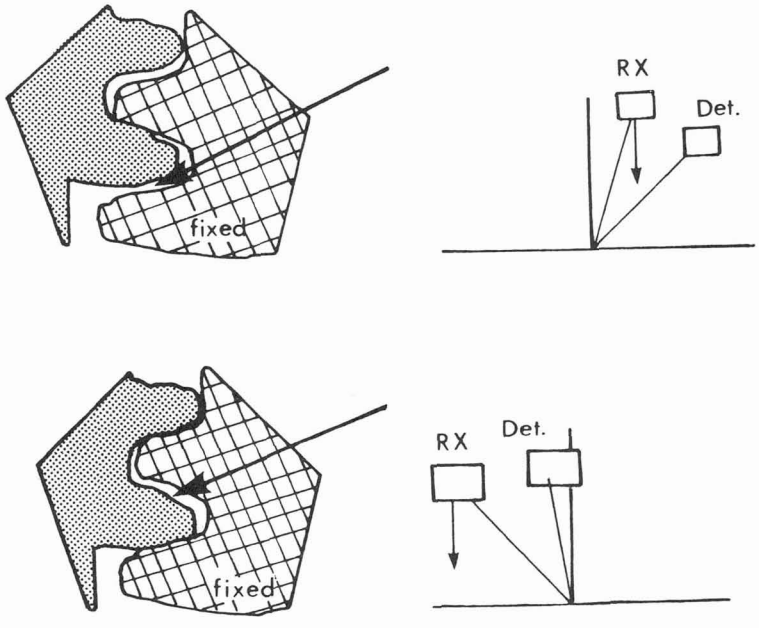

Figure 3a, 3b : Representation of the error induced by the mechanical plays which exist between the pinion and the disk according to the X-ray tube/detector position by reference to a vertical axis.

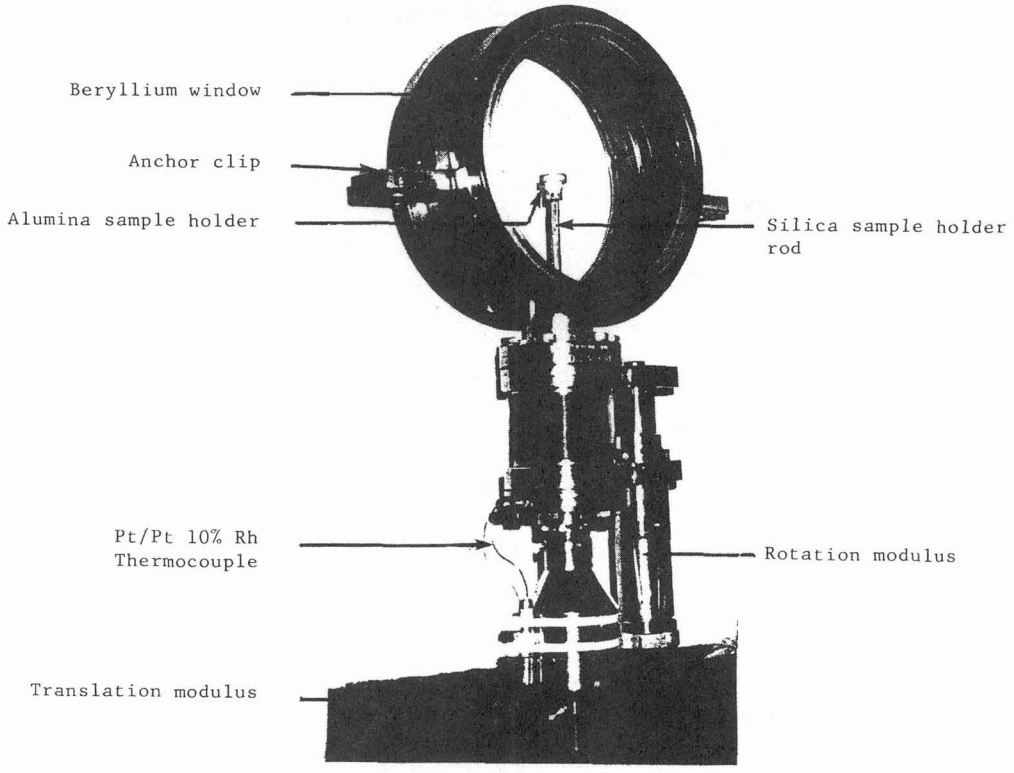

Figure 4 : A central part description.

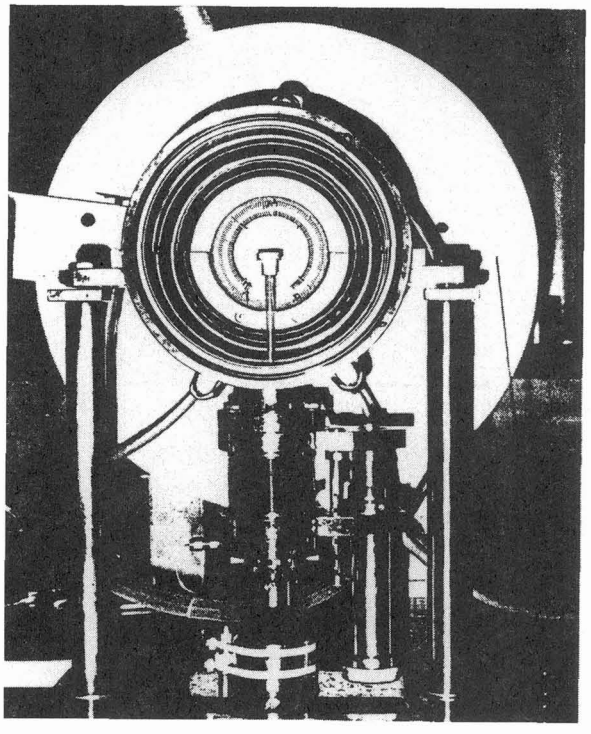

Figure 5 : High temperature chamber : inner view.

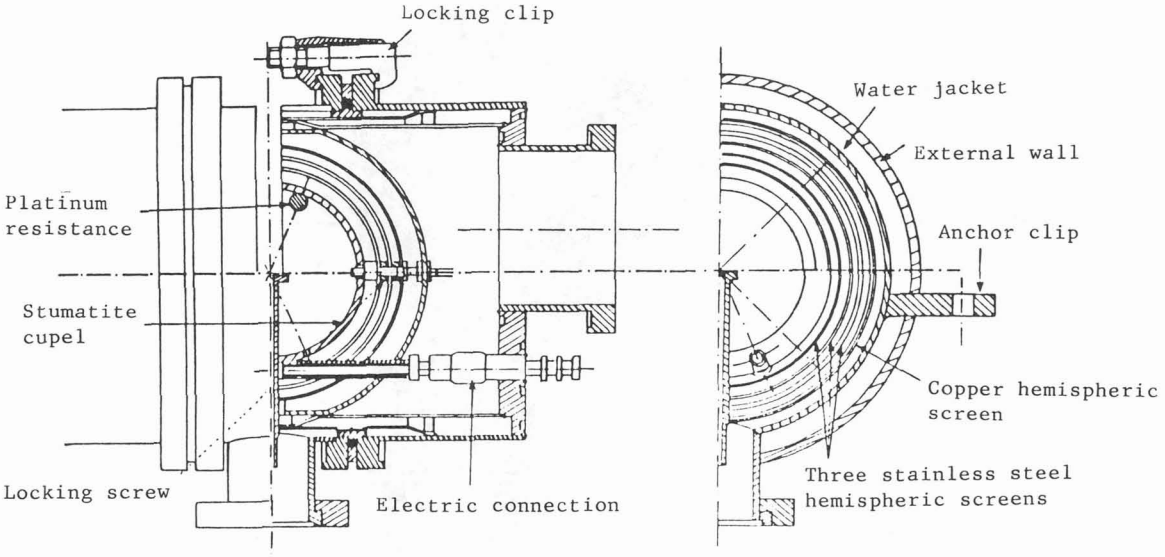

Figure 6 : Cross section of the chamber.

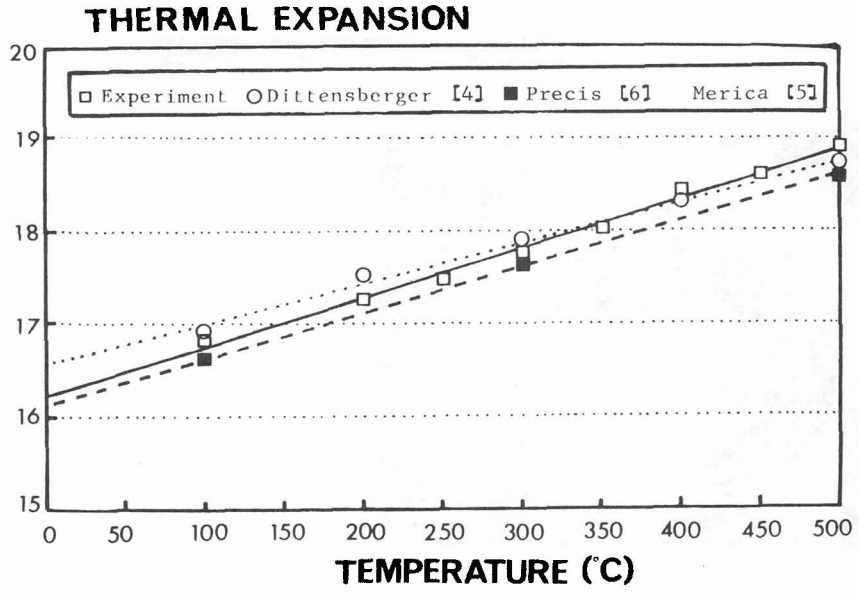

Figure 7 : Evolution of the thermal expansion for a copper plate versus temperature under a vacuum.