EXPERIMENTAL ANALYSIS OF RESIDUAL STRESSES BY X-RAY DIFFRATOMETRY

Method not yet standardized.

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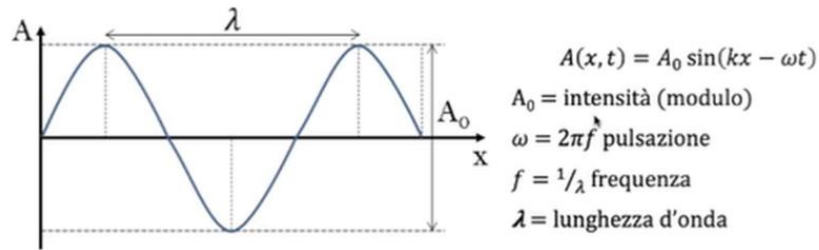
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X-Ray Diffraction

Introduction

It is a non-destructive technique that allows to measure the residual stresses on the "surface" of materials that have a crystalline lattice for which: metal and ceramic. The principle of operation is based on the measurement of lattice distortion to solve deformations and stresses.

The advantages of diffractometry are:

- Non-destructive measurement;
- Measurement of non contact;
- Non-field measurement: I can get the voltage state at a point;
- The surface does not need any preparation;
- Can be combined with electropolishing but becoming a destructive method;

the disadvantages are:

- No field measurements carried out;
- Limited to crystalline materials;
- Needs X-ray source

Experimental Setup

The measuring system consists of an omega headband with photodetector and a collimator that directs X-rays on the surface of the materials under consideration.


The entire arch is mounted on a mechanical arm that can lift and lower, the bow instead can rotate clockwise and counterclockwise, later you will see the usefulness of such features.

Physical Principle Diffraction

We introduce some physical quantities of electromagnetic waves, this can be indicated as:

$$A(x,t) = A_0 \sin(kx - \omega t)$$
A_0 = intensità (modulo)
$\omega = 2\pi f$ pulsazione
$f = 1/\lambda$ frequenza
λ = lunghezza d'onda

Suppose we have a spherical body of diameter d, and it is hit by a wave of wavelength λ, it will become a secondary emitter and will emit waves at the same λ.
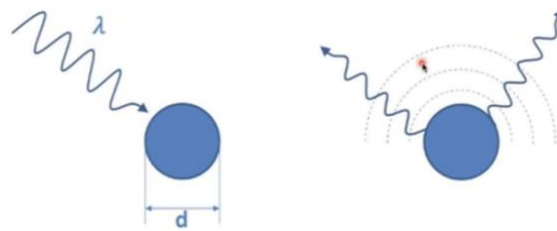
Two waves with the same wavelength can interfere with each other, the laser swords of different color of star wars that interfere are a fiction.

Therefore, suppose we have two waves with the same wavelength but spaced between them by a temporal δ amount:
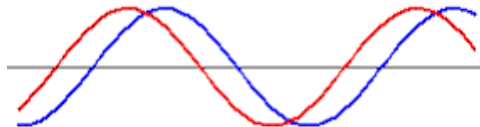
they will be:

$$A_1(t) = A_0 \sin(kx - \omega t)$$

$$A_2(t) = A_0 \sin(kx - \omega t + \delta)$$

The intensity resulting from the interference:

$$A_{tot}(t) = 2A_0 \cos(\delta/2)$$

Depending on the δ I can have:

$$\begin{cases} \delta = 2n\pi \rightarrow constructive\ interference \rightarrow A_{tot} = 2A_0 \\ \delta = 2n\pi \rightarrow destructive\ interference \rightarrow A_{tot} = 0 \end{cases}$$

Simple Ideal Lattice

Consider two spherical sources S_1,S_2 and an observer O. The two sources are located at a distance from each other and emit waves of the same wavelength, but the path to A_1 to reach O will be different from the path to be covered by A_2, which will therefore be delayed by δ:
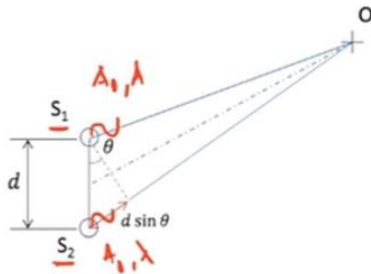
$$S_1 O = path\ of\ A_1$$

$$S_2 O \cong S_1 O + d * \sin(\theta) = path\ of\ A_2$$

therefore $\delta \propto d\sin(\theta)$ in fact:

$$\omega : \delta = c : d\sin(\theta)$$

$$c = wave\ propagation\ velocity$$

from which we get:

$$\delta = \frac{\omega}{c}\ d\sin(\theta) = \frac{2\pi f}{c}\ d\sin(\theta) = \frac{2\pi}{\lambda}\ d\sin(\theta)$$

The type of interference, fixed λ (depending on the X-ray tube), depends on sin (θ):

$$\delta = \frac{2\pi}{\lambda}\ d\sin(\theta)$$

Ideal Crystal Lattice

Now consider a crystalline lattice of a material, in the absence of loads, TR and microstructural defects something of the following type happens, where, each atom is spaced by d_0 from the other and will act as emitter:
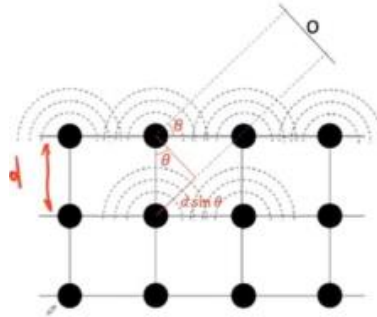

The δ required for a reading of the instrument is that which produces me constructive interference, and therefore the maximum intensity perceptible:

$$\begin{cases} \delta = \dfrac{2\pi d}{\lambda} d \sin(\theta) \\ \delta = 2n\pi \end{cases} \to n\lambda = d \sin(\theta)$$

This is the Law of Bragg or Fundamental Law of Diffractometry.

The signal detected by the photodetectors, distributed along the head of the measuring head, will be of the following shape. This is because each secondary emitter emits spherical waves that interfere with each other and there is a direction θ such that the intensity is maximum and is characteristic of each material:
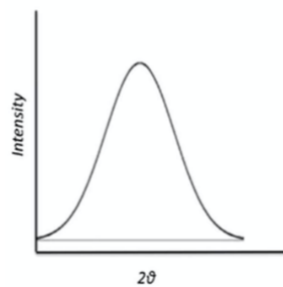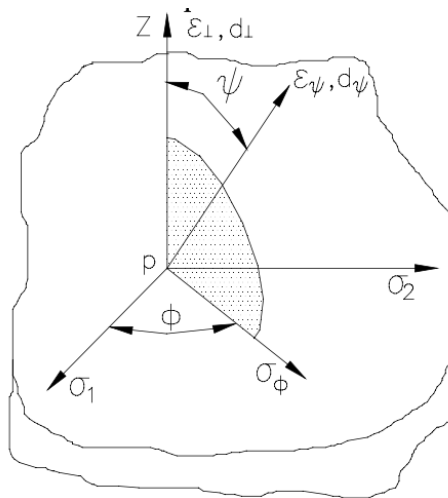
Ideal Crystalline Lattice Loaded

But if I load the sample, for example, I strain it, the atoms move away in the direction of stress, but they approach the surface in the normal direction because the cross section is reduced:

$$d \downarrow \rightarrow since\ n\lambda = cost \rightarrow \theta\ must\ increase \rightarrow SHIFT\ of\ the\ signal$$
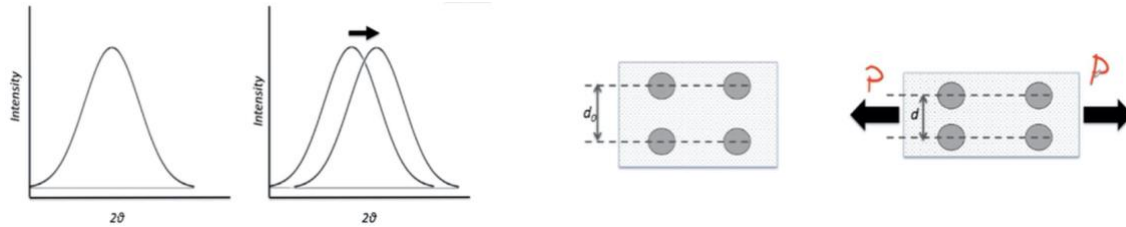
Strain

Let's take a reference system attached to the surface:

Suppose we want to measure the deformation along the z-axis normal to the surface of the piece, we define ε_z as a variation of the interatomic distance:

$$\epsilon_z = \frac{d_z - d_0}{d_0}$$

X-rays are used because they have a wavelength that can interfere with the interatomic distances of crystalline materials.

The tension then in case of **plane stress**:

$$\epsilon_z = \sigma_z - \frac{\upsilon}{E}(\sigma_x + \sigma_y), \sigma_z = 0$$

$$\frac{d_z - d_0}{d_0} = -\frac{\upsilon}{E}(\sigma_x + \sigma_y)$$

Knowing the properties of the material (υ,E), I measure d_n with diffractometry. The only unknowns are σ_x, σ_y e d_0.
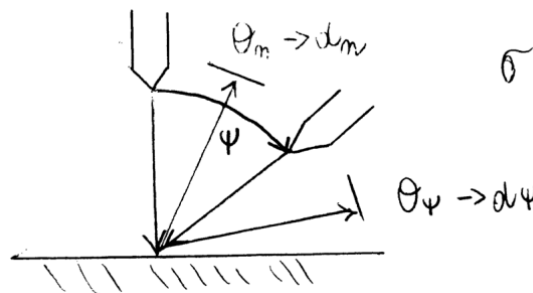
The problem is that d_0 of an alloy with random grain arrangement is not known a priori, also there is no material that is TR free against a processing. Also, I need at least two measures to discriminate separately σ_x e σ_y.

Usually, we don't deal with **monoscristalline** structures, and each crystal can have different directions of growth, I can use a more general reference system where each crystalline grain will contribute according to its own direction of growth:

$$\epsilon_{\phi,\psi} = \frac{d_{\phi,\psi} - d_n}{d_n}$$

What is actually done is to place the nominal collimator on the surface and measure for θn from which I derive d_n; I then rotate the collimator ψ and gain θ_ψ and d_ψ; therefore, with the **Law of Bragg**:

$$\begin{cases} \psi = 0 \rightarrow \max \theta_n \rightarrow \theta_n \rightarrow n\lambda = d_n \sin(\theta_n) \rightarrow d_n \\ \psi \neq 0 \rightarrow \max \theta_\psi \rightarrow \theta_\psi \rightarrow n\lambda = d_\psi \sin(\theta_\psi) \rightarrow d_\psi \end{cases}$$


And you can thus calculate the voltage with the following:

$$\sigma_\psi = \frac{E}{(1+v)\sin^2(\psi)} \frac{d_\psi - d_n}{d}$$

From the theoretical point of view, only two measures are enough, from the practical point of view instead: up to five measures are made with ψ_i different because with only two measures could be measured surface tensions altered by an inclusion "close" to the surface.

A linear regression line of the results is then made and the voltage expressed as a function of this line:
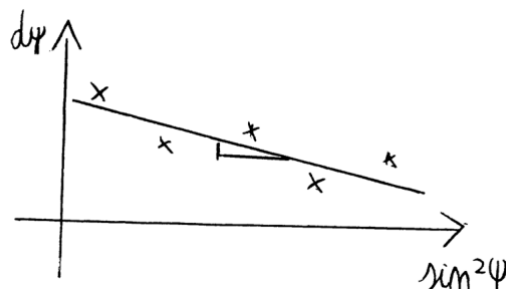
And it is calculated:

$$\sigma_\psi = \frac{E}{(1+v)\sin^2(\psi)} \frac{d_\psi - d_n}{d_n} = \sigma_\psi = \frac{E}{(1+v)\sin^2(\psi)} \frac{d\psi}{} = \frac{E}{(1+v)} m$$

Effect of the plastic shear deformation

Imagine having a RS-free material and applying a cutting load to it that causes plastic shear deformation. Such deformation would lead to a shift of the crystalline planes but the distance between them would remain unchanged. What happens is that based on how I position the collimator, I get residual **apparent** tensile or compressive stresses:
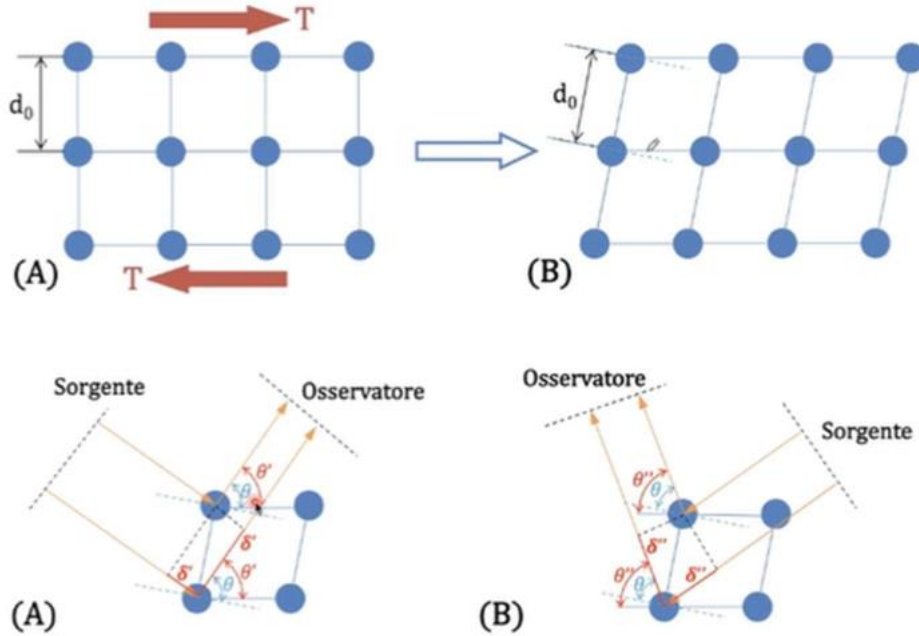
If I place the collimator on the left, $\theta' < \theta \rightarrow$ if $\theta \downarrow$ then $d \uparrow \rightarrow$ TR apparent traction.

If I place the collimator on the right, $\theta' > \theta \rightarrow$ if $\theta \uparrow$ then $d \downarrow \rightarrow$ TR apparent compression.

To overcome this problem, I tilt the collimator to the right and left of the same angles, I find the bisector between the two linear regressions (regression of the right angles and regression of the left corners):
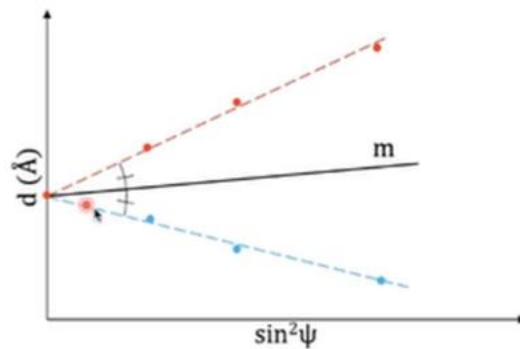
I use the bisector to characterize the stress:

$$\sigma_\psi = \frac{E}{(1 + v)} m$$

Diffractometry + Elettropolishing

I can combine what was previously seen with the EDM that does **not introduce** Resisal Stresses to study the tensional state in depth, i.e.:

- I perform the measurements;
- Remove a certain amount of material;
- Repeat measurements on new surface;
- I repeat the previous steps.

We have three possible areas of work in the electrolytic removal and the amount of material removed is proportional to the current, that is:
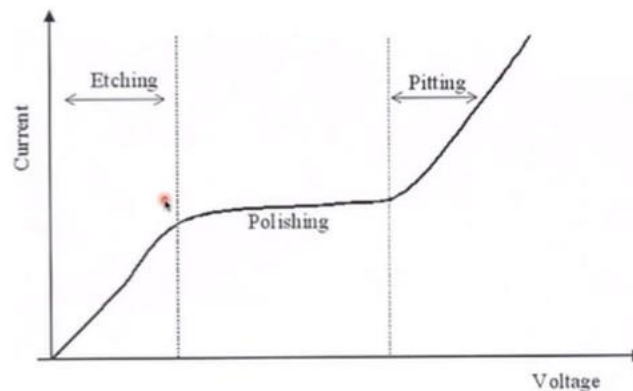
In a first zone there is **Etching** (this phase is generally used for polishing). The second phase is that of our interest, the reason is linked to the fact that you can remove in a controlled manner. In the third one you have discharges; you don't work in this area.

Advantages over the hole method:

- Theoretically I have no drilling limits. Remember that in hole drilling you get up to a maximum value of 1-2 mm. The limit is that rosettes are not sensitive beyond a certain value.
- I can vary the steps; this is useful for example in the case of shot peening. In the case of shot peening, there are very high gradients.

Measurement Errors

- Errors related to instrumentation;
- Errors due to the imperfection of the specimen: the surfaces of the specimen can either plant or have a curvature. In case these have curvature, this must be limited, as if it were not, I would be inaccurate. What happens is that the radius of curvature must be at most 2 times the diameter of the collimator.
- Errors due to peak-fitting: that is, the signals, do not have an ideal Gaussian pattern but have a noise;