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Nondestructive Characterization of Materials IV

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PREFACE

There is a great deal of interest in extending nondestructive technologies beyond the location and identification of cracks and voids. Specifically there is growing interest in the application of nondestructive evaluation (NDE) to the measurement of physical and mechanical properties of materials. The measurement of materials properties is often referred to as materials characterization; thus nondestructive techniques applied to characterization become nondestructive characterization (NDC).

There are a number of meetings, proceedings and journals focused upon nondestructive technologies and the detection and identification of cracks and voids. However, the series of symposia, of which these proceedings represent the fourth, are the only meetings uniquely focused upon nondestructive characterization. Moreover, these symposia are especially concerned with stimulating communication between the materials, mechanical and manufacturing engineer and the NDE technology oriented engineer and scientist. These symposia recognize that it is the welding of these areas of expertise that is necessary for practical development and application of NDC technology to measurements of components for in-service life time and sensor technology for intelligent processing of materials.

These proceedings are from the fourth international symposia and are edited by C.O. Ruud, J. F. Bussiere and R.E. Green, Jr. The dates, places, etc of the symposia held to date area as follows:

TITLE:	Symposia on Nondestructive Methods for Material Property Determination
DATES:	April 6-8, 1983
PLACE:	Hershey, PA, USA
CHAIRPERSONS:	C.O. Ruud and R.E. Green, Jr.

TITLE:	Second International Symposia on the Nondestructive Characterization of Materials
DATES:	July 21-23, 1986
PLACE:	Montreal, Quebec, CANADA
CHAIRPERSONS:	J.F. Bussiere, R.E. Green, Jr., J.P. Mouchalin and C.O. Ruud

TITLE: Third International Symposium on Nondestructive Characterization of Materials
DATES: October 3-6, 1988
PLACE: Saarbrucken, Germany
CHAIRPERSONS: P. Holler, R.E. Green, Jr. and C.O. Ruud

TITLE: Fourth International Symposium on the Nondestructive Characterization of Materials
DATES: June 11-14, 1990
PLACE: Annapolis, MD, USA
CHAIRPERSONS: R.E. Green, Jr. and C.O. Ruud

The title, editors, etc of the proceedings of these symposia are as follows:

TITLE: Nondestructive Methods for Material Property Determination
EDITORS: C.O. Ruud and R.E. Green, Jr.
PUBLISHER: Plenum Press, New York, NY, USA
YEAR PUBLISHED: 1984

TITLE: Nondestructive Characterization of Materials II
EDITORS: J.F. Bussiere, R.E. Green, Jr., J.P. Mouchalin and C.O. Ruud
PUBLISHER: Plenum Press, New York, NY, USA
YEAR PUBLISHED: 1987

TITLE: Nondestructive Characterization of Materials
EDITORS: P. Holler, V. Hauk, G. Dobmann, C.O. Ruud and R.E. Green, Jr.
PUBLISHER: Springler-Verlag, Berlin
YEAR PUBLISHED: 1989

TITLE: Nondestructive Characterization of Materials IV
EDITOR: C.O. Ruud, J.F. Bussiere and R.E. Green, Jr.
PUBLISHER: Plenum Press, New York, NY, USA
YEAR PUBLISHED: 1991

The fifth International Symposium on Nondestructive Characterization of Materials was held May 27-30, 1991 in Karuizawa, Japan and the proceedings are in preparation at this printing.

CONTENTS

COATINGS AND THIN FILMS

WOOD, PAPER, CONCRETE, AND CERAMICS

MATERIAL CHARACTERIZATION AND MODELING

MICROSTRUCTURE AND PLASTIC DEFORMATION OF METALS

COMPOSITES

ACCURATE STRUCTURAL CHARACTERIZATION OF ZrN COATINGS AND EPITAXIAL LAYERS BY X-RAY DIFFRACTION USING THE DOSOPHATEX SYSTEM

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INTRODUCTION

Thin film characterization by X-Ray methods is often difficult because of the problems of preferred crystal orientations and residual stresses. For example, it is well known that coatings made by physical vapor deposition are very strongly textured so that measurements of residual stress by classical X-Ray methods are very inaccurate. Furthermore, the characterization of epitaxial thin layers of semi-conductors requires very accurate determination of crystal orientations and interfacial misfits.

A new diffractometer[1] has been developed to form a multipurpose 4-circle assembly (α, 2θ, ϕ, ψ), the different rotational positions of which are selected by computer driven high precision and high speed stepping motors (Fig. 1). Depending on the type of angular scanning selected, as well as the data processing and acquisition software, the following analysis may be rapidly performed on our system :

* Identification of textured phases,
* Volume fraction determination,
* Texture analysis by pole figures and Orientation Distribution function (ODF),
* Single crystal, or epitaxial layer orientations,
* Residual stress measurements (by psi setup),
* Grazing X-ray analysis of ultra-thin layers.

In fact these analyses complement each other and enable us to undertake any type of complex research. Here we shall present a few applications.

A- PHASE IDENTIFICATION

This is a basic analysis for materials characterization, which must precede all other analyses and must be perfect : all the peaks {hkl} of all the existing phases of the sample must be detected. Usually the well-known disturbing effects of texture lead to less accurate results.

In standard analyses of a single crystal of {111} oriented silicon, only the {111} peak and its multiples are detected (Fig. 2).

Nondestructive Characterization of Materials IV
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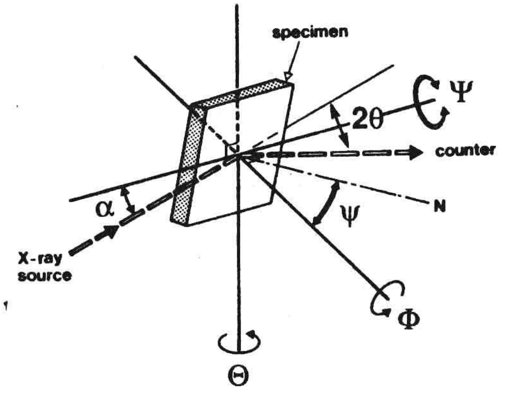

Fig. 1. The 4 circle assembly (α, 2θ, ϕ, Ψ) of the Dosophatex system.

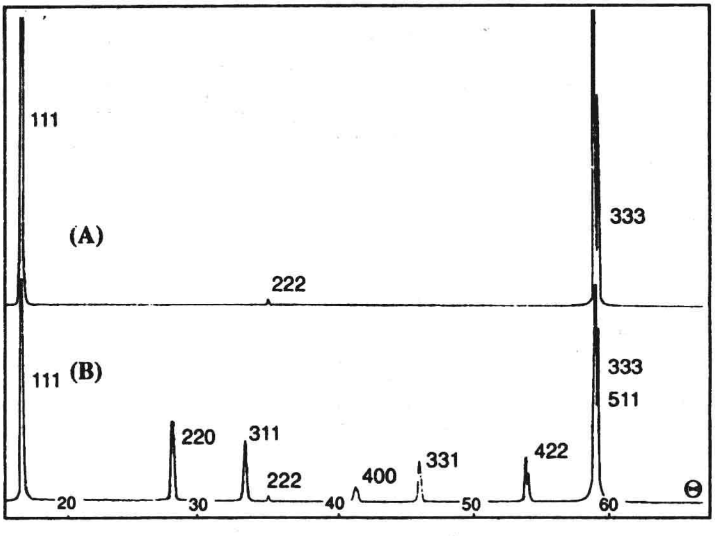

Fig. 2. θ-2θ diagrams of a single crystal of Silicon oriented (111)
(A) : Standard analysis.
(B) : Dosophatex analysis which performs integration over the range ϕ (0°-360°) and Ψ (± 60°).

On the other hand, using our system, due to a rapid sample rotation and oscillation, it is rendered isotropic with respect to the analysis : all the {hkl} peaks appear.

One of the main advantages of our system consists in the analysis of new phases[2].

B- QUANTITATIVE ANALYSIS

Determining the volume fraction of the phases of a multi-phased material is fundamental for knowledge of its strength properties or resistance to corrosion for example. With standard analyses, the effects of texture, which are almost always present, render quantitative phase analysis very difficult and rarely performed. But, with our system quantitative analysis is a frequent application.

Dosophatex enables one to solve problems as complex as determining the volume fraction of monoclinic and tetragonal zirconia in zirconia-toughened alumina ceramics[3]. This quantitative analysis is fundamental to understand toughening mechanisms. Dosophatex analysis is a general method capable of performing volume fraction determination of α, γ and carbides phases in high-speed steels where 6 to 7 phases are associated, while producing results to within 1 % ! Of course phase structure must be perfectly known in order to calculate theoretical intensities in connection with geometry assembly and for the wavelength used.

C - STRESS DETERMINATION

We shall present the possibilities our system offers by studying ZrN film deposited to a thickness of 5 µm onto stainless steel then annealed at 900° 1 hour[4].

In order to avoid the problem of an evolution in the composition with respect to the depth analysis in the deposit, we selected a deposit offering the best possible homogeneity, by means of preliminary Auger and electron probes[5,6] analysis.

The phase analysis was performed,using Cobalt Kα radiation, in order to select all the ZrN peaks {hkl} which did not overlap those of the austenitic steel substrate (Fig.3).

Moreover, for deformation measurements, we selected Chromium Kα radiation to limit as much as possible the analysis depth. We observe linear strain versus $\sin^2\phi$ plots (sspp) on each plane considered (Fig.4) showing a strong biaxial compressive stress on the surface of the deposit. To calculate the stress, we used a Young's modulus, of 460,000 MPa, and a Poisson's ratio 0.2. Also, since the X-ray elastic anisotropy was not known, we attempted to determine it directly : the measurement must give the same stress value, regardless of the {hkl} peak considered, if we enter the correct X-ray elastic anisotropy into the calculation[7,8].

The compressive stresses for the different planes considered are calculated by varying the X-ray elastic anisotropy (A_{xc}) (Fig.5). The curves roughly intersect at the same point, which according to the least square error method, gives us for A_{xc} the value of 0.83. By taking this A_{xc} value, we find that the surface compressive stress is σ = -7,000 MPa.

On the other hand, with more penetrating Copper Kα radiation passing entirely through the deposit, the deformation curves are no longer linear (Fig.6) and demonstrate that shear stresses exist[9]. This is directly linked to the stress gradient through the deposit, which may be explained by the different cooling rates between the substrate and the deposit.

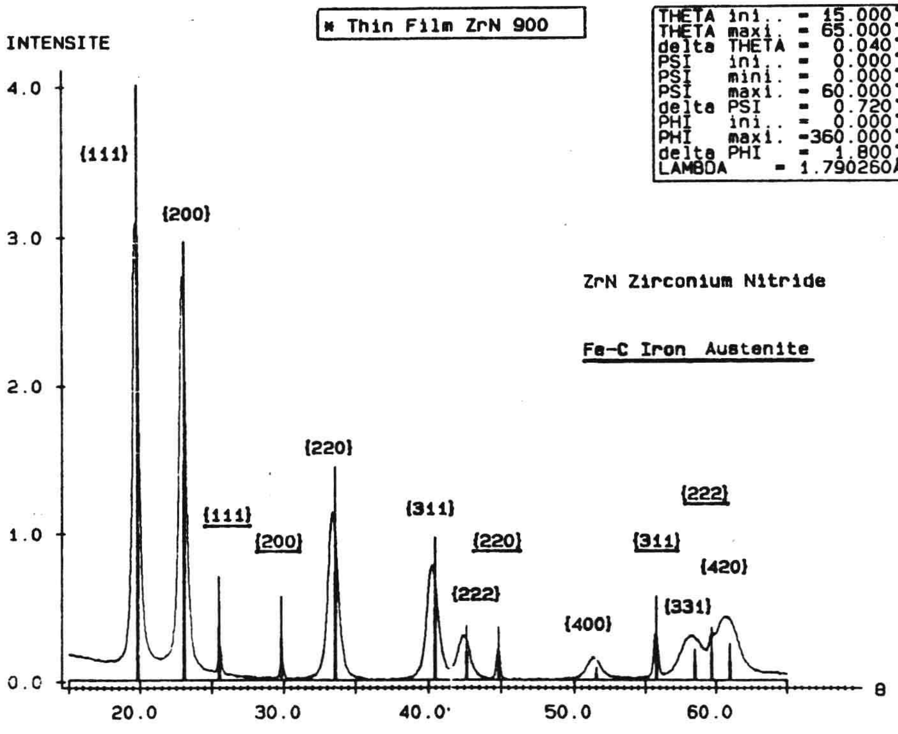

Fig. 3. XRD diagram with CoK_α for ZrN sample using Dosophatex system.

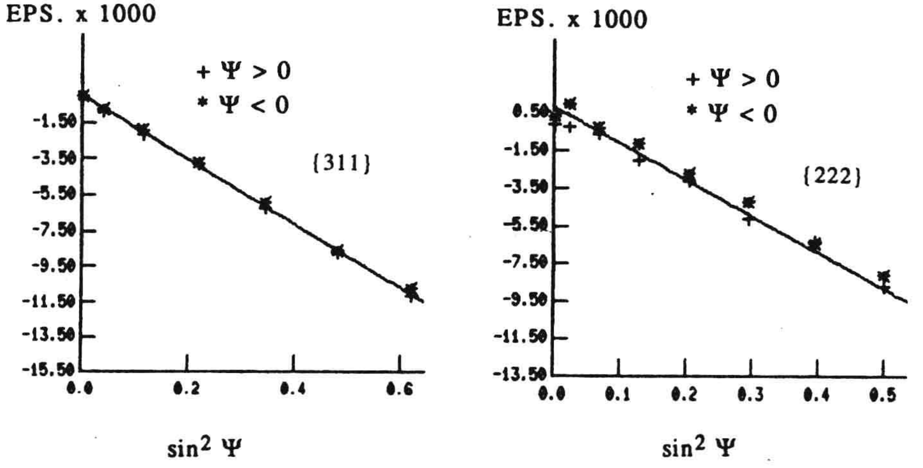

Fig. 4. Plots of residual strain as a function of $\sin^2 \Psi$ from {311} and {222} diffraction peaks of ZrN deposit as determined with $K_\alpha Cr$.

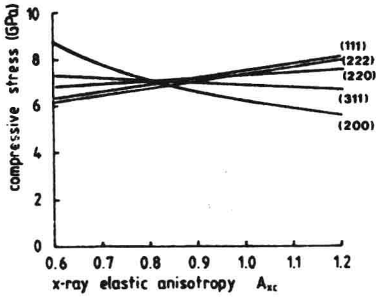

Fig. 5. Residual stress as a function of the ration $A_{xc} = \frac{S_2(h00)}{S_2(hhh)}$

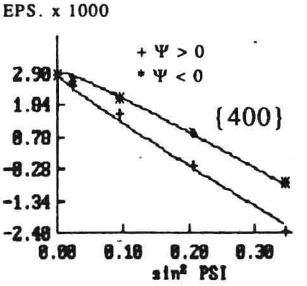

Fig. 6. sspp from {400} CuK$_\alpha$ diffraction peak of the ZrN film

D- TEXTURE ANALYSIS

Until now, we have considered textures with respect to their negative aspects on the accuracy of results, and we have presented a way of minimizing these negative effects.

However, it is equally essential to perfectly determine these textures in order, for example, to calculate the behavior of materials that are submitted to mechanical deformations.

We shall consider an extreme case of analysing epitaxial layers for the manufacture of photo-voltaïc cells[10].
Two types of layers were analysed :

(1) ZnSe layers on (100) oriented GaAs single crystal substrates. The purpose was to develop epitaxy via their isomorphism, which differs only by 0.3 %. Note, however, that ZnSe also crystallizes to a metastable hexagonal phase[11]

(2) GaAs layers on Ge single crystal substrate oriented (100). The isomorphism in this case is identical to within 0.07 %.

These layers were developed according to the close space vapor transport method (CSVT)[12]. This method is simple, low cost and offers the advantage of a high yield capable of reaching 90 %. We select two ZnSe layers through their morphology using SEM

(1) layer #1 was a non-bonding deposit and spontaneous debonding occured ; the interface between the layer and the substrat was smooth.

(2) layer #2 deposit was a highly bonding deposit.

Now, it is important to verify epitaxial relations, if they exist. In general, epitaxy is checked by means of Rocking Curves, which is a very high accuracy method, but which is used only to check simple epitaxial relations[13,14].

For more complex cases, with possibility of multiple epitaxial phases, the pole figure still remains the most efficient means for determining crystallographic orientation relations between the substrate and the deposit. For this operation, we developed an original method using high-resolution ultra-rapid pole figures (up to ψ = 80°) for which we performed the

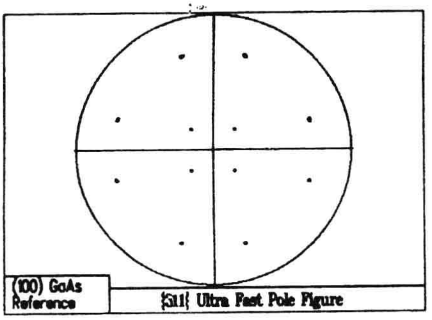

Fig. 7. High resolution {311} pole figure of a reference GaAs (100) single crystal

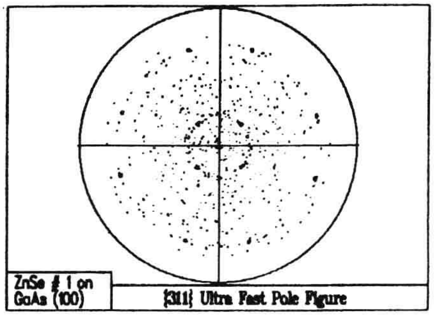

Fig. 8. High resolution {311} figure of layer ZnSe #1

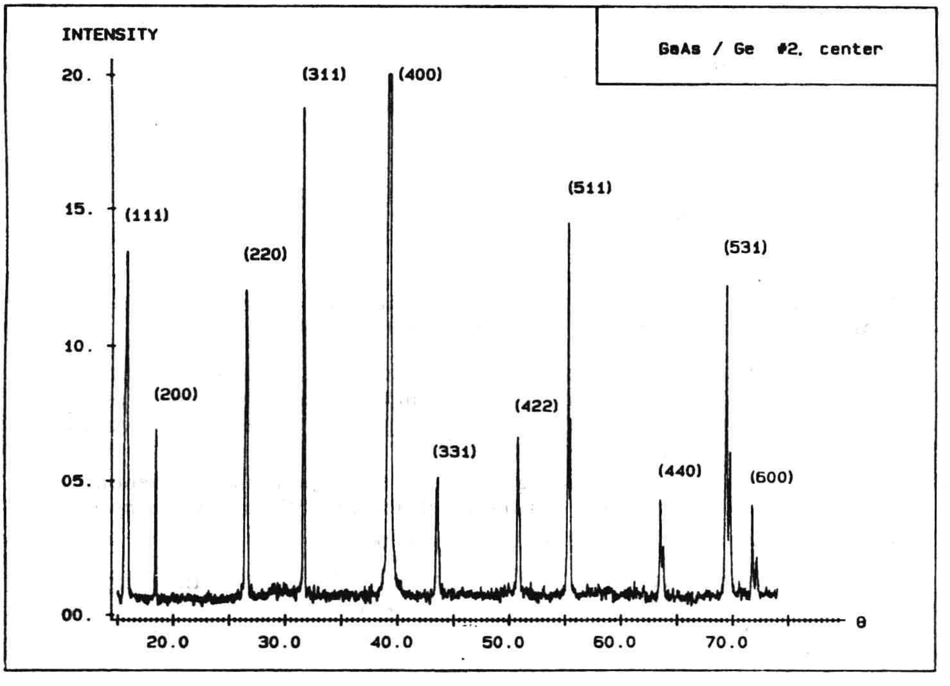

Fig. 9 . XRD Dosophatex diagram of layer GaAs # 2