World Academy of Science, Engineering and Technology
International Journal of Mechanical and Mechatronics Engineering
Vol:8, No:2, 2014

Development of Material Analyzing Software Using X-Ray Diffraction

Le Chi Cuong

***Abstract*—**X-ray diffraction is an effective mean for analyzing material properties. This paper developed a new computational software for determining the properties of crystalline materials such as elastic constants, residual stresses, surface hardness, phase components, and etc. The results computed from the X-ray diffraction method were compared to those from the traditional methods and they are in the 95% confidential limits, showing that the newly developed software has high reproducibility, opening a possibility of its commercialization.

***Keywords*—**X-ray diffraction, Nondestructive evaluation, Hardness, Residual stress, Phase determination.

I. Introduction

AMONG many methods of material analyzing as magnetic, ultrasonic [1], microscopic [2], laser, X-ray diffraction methods [1], [3] etc.; X-ray diffraction method has many advantages over the other methods, because it can nondestructively and accurately determine the material properties such as residual stress [4], [5], crystalline grain size [6], phase quantitative [7], hardness [8], thin layer thickness [9] etc. A previous research has developed automated computational software on stress of polycrystalline material [10]. However, many computations on the correction for absorption factor for various materials having different have not been totally integrated into the software.

This research develops a new X-ray material analyzing software for determining material properties, called X-Pro 1.0. The programming language used was C#, which is a strong language, widely supported by the programming experts. It is also capable for users having little programming experience to revise or add the source for a proper computation.

II. Material Analyzing Software X-Pro 1.0

A. Selection of Development Language

Two common programming methods are currently used, including structured programming (SP) and object-oriented programming (OOP). The SP languages, which are Assembly, Basic, C, and Pascal, have common features such as "*Program = Data structure + Algorithm*" [11]. Besides the advantage of ease to follow, SP has disadvantages that it does not allow to reuse the resource code, the algorithm strictly depends on the data structure; as the structure changes, the algorithm must change, thus make it difficult to develop, especially for a large project. Instead, OOP uses a group of functions and variables to solve the task of the objects, thus save the resources, reuse the code and is suitable for a large project. Among the OOP languages as Turbo Pascal, C++, and C#, C# is a strong language, developed from the .NET Framework background and supporting many libraries for utilizing rapid programming [12], [13]. The newly developed computational software X-Pro 1.0 was programmed using C# has functions of analyzing material properties as stress computation, phase quantitative determination.

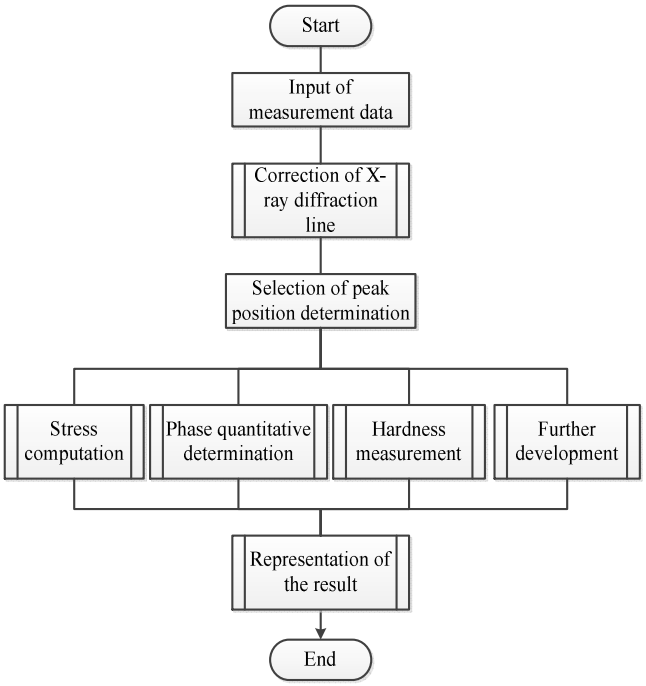

Fig. 1 Flow chart of X-Pro 1.0

B. Data analysis

The measurement data files are read from the X-ray diffraction system Panalytical XPert or from a text file and then plotted into the diffraction lines as shown in Fig. 2. The peak position *p* and line broadness *B* is shown directly on the diffraction line for preliminary evaluation. The absorption factor of many measurement methods used for correcting X-ray intensities are listed in Table I. The diffraction planes (*hkl*) are also analyzed together with the wave length and the type of Bravais lattice of the measured material. Table I shows the absorption factor for correcting the X-ray intensity in the case of measuring many diffraction methods.

L. C. Cuong is in Faculty of Mechanical Engineering, the University of Technical Education-HCMC, 01 Vo Van Ngan Str., Thu Duc Dist., HCMC, Vietnam (phone: 84 8 37221223; fax: 84 8 38964922; e-mail: lccuong@hcmute.edu.vn).

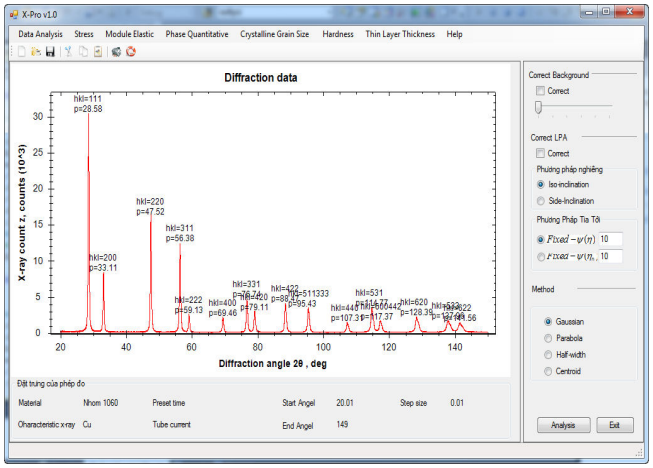

Fig. 2 Main window of data analysis

TABLE I
ABSORPTION FACTORS FOR ISO-INCLINATION AND SIDE-INCLINATION METHODS [14]

Inclination method	Fixed-	Limination of irradiated area	
		Without	With
Iso-	ψ_0	$1-\cot(\theta-\psi_0)\cot\theta$	$\cos\psi_0[1-\cot(\theta-\psi_0)\cot\theta]$
	ψ	$1-\tan\psi\cot\theta$	$\sin(\psi+\theta)[1-\tan\psi\cot\theta]$
Side-	η_0	$1+\tan(\theta-\theta_0)\cot\theta$	$\cos\varphi\sin\theta_0[1+\tan(\theta-\theta_0)\cot\theta]$
	η	1	$\cos\varphi\sin\theta$

C. Smoothing

To smooth the rough measured X-ray counts, the data is calculated from three data (x_1,y_1), (x_i,y_i), and (x_2,y_2), having an internal of nc, where n is an integer and c is the step size. The slope of the line (1, 2) is:

$$\tan\alpha=\frac{y_2-y_1}{x_2-x_1} \quad (1)$$

For most normal measurement, the angle α is preset at 10° to get distinction between the diffraction peak and the background diffraction. The value of n can be preset from 1 to 10 to change the smoothing level. Fig. 2 is the diffraction line for CeO_2.

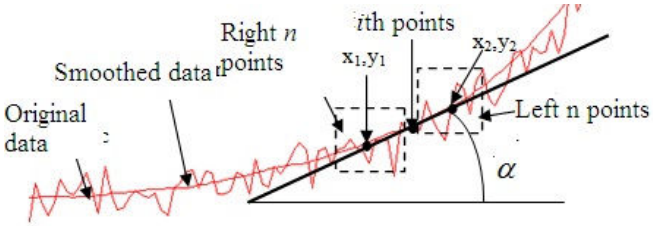

Fig. 3 Determination of slop in smoothing technique

D. Stress Determination

The stresses, both residual stresses and loading stresses, on the surface of crystalline materials can be determined by X-ray diffraction measurement data. The dialog box shown in Fig. 4 allows calculating the material elastic constants as Young's modulus and Poisson ratio used for stress computation. This also makes it possible to revise a material or add a newly measured material and then save to the program library. By clicking "OK", the dialog box for selecting methods of peak position determination, the correction factors used for stress computation is shown as Fig. 5. The corrected X-ray diffraction line, peak positions, stress together with their 95% confidential limits representing the reproducibility of the calculated value are shown.

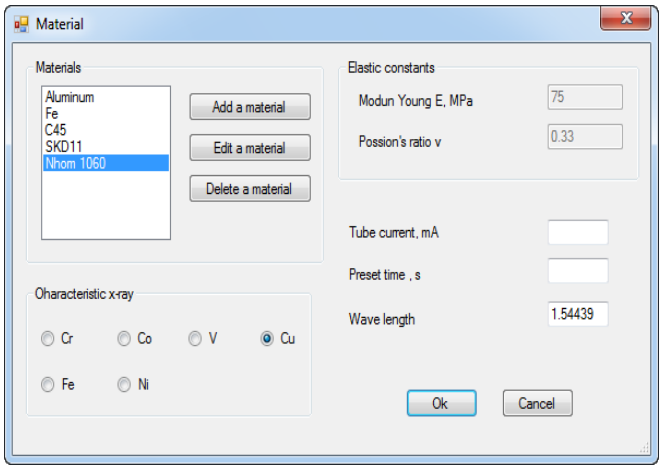

Fig. 4 Dialog box for computing elastic constants

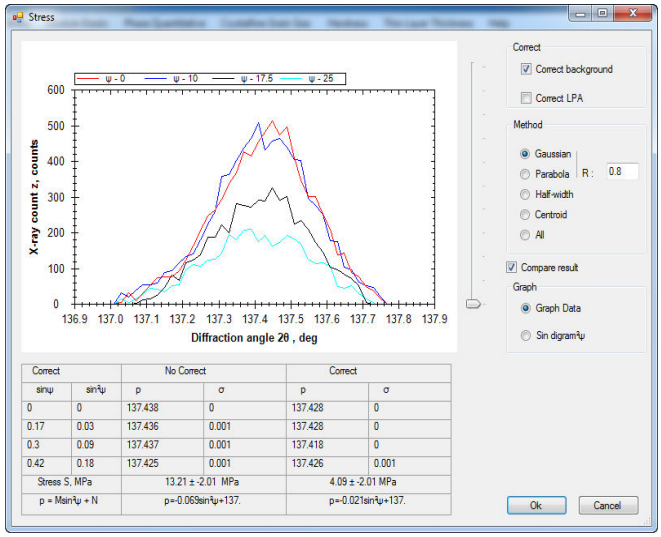

Fig. 5 Dialog box of stress determination

Four common methods for stress calculation including the Gaussian curve, the parabola, the half-width and the centroid methods [5]. In the Gaussian curve and parabola methods, the peak positions are determined by fitting a curve to the measurement data points above R% of the maximum X-ray counts. The centroid method determines the peak position from the integral area under the diffraction line, so it strongly depends on the scanning, thus lead to low accuracy of computation. In the half-width method, the peak positions are determined from six data points around half of maximum X-ray counts.

TABLE II
RESIDUAL STRESS OF ALUMINA ALLOY 1060

Zones	X-Pro 1.0 (MPa)				Origin Pro 8.5.1
	Gaussian	Parabola	Half width	Centroid	Gaussian
1	-17.3 ± 2.8	-17.3 ± 0.2	5.4 ± 19.7	7.5 ± 9.4	- 25.3
2	-26.0 ± 8.9	26.0 ± 0.4	-3.2 ± 18.3	-2.4 ± 7.2	-30.4
3	13.2 ± 3.1	13.2 ± 0.4	11.6 ± 42.8	8.7 ± 8.5	10.1
4	-29.5 ± 6.0	-29.4 ± 0.6	-19.8 ± 27.7	-23 ± 9.6	-28.3

Table II shows the residual stresses in a butt-weld of aluminum alloy 1060 using the friction stir welding technique, calculated from XPro v1.0 by using four methods of peak position determination: Parabola, Gaussian, Half-Width, Centroid, together with 95% confidential limits [15]. It is observed that the Gaussian curve and parabola methods give closer value than the other methods. On the other hand, the half-width and the centroid methods have large variation of the calculated value. This shows that the Gaussian curve and parabola methods should be used for stress determination using X-ray diffraction.

E. Phase Quantitative Determination

X-Pro 1.0 can determine the phase component for double phase materials. Choosing "Phase quantitative" on the menu bar, then choosing "Open file", the dialog box as indicated in Fig. 6 for selecting diffraction peak for each phase is shown. The diffraction parameters for determining diffraction plane such as wave length, lattice type and parameters are shown.

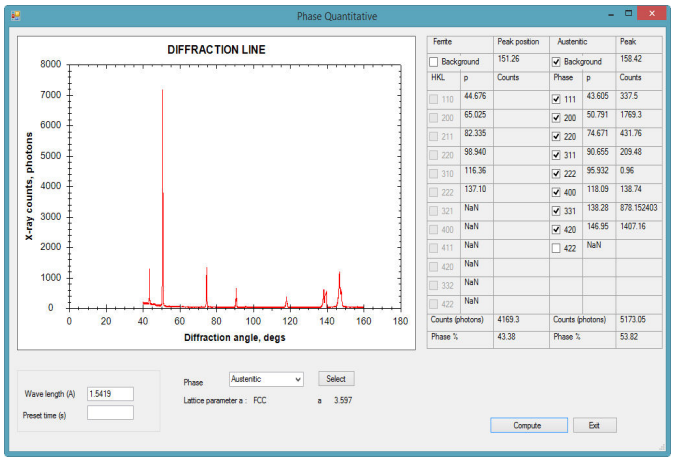

Fig. 6 Dialog box for phase quantitative determination

In the case of high background diffraction of more than 10% of maximum X-ray intensity, background should be subtracted. Otherwise, the background diffraction should be treated as extraneous matter component. In those cases, the button "Background separation" should be chosen for each phase.

Choosing "Phase Quantitative Determination" on the menu bar, the dialog box for phase determination is shown as in Fig. 6. The method of computation has been published as [7]:

$$q_\alpha = \frac{\sum E^{\alpha}_{(hkl)^{\alpha}_i \lambda_j}}{\sum E^{\alpha}_{(hkl)^{\alpha}_i \lambda_j} + \sum E^{\gamma}_{(hkl)^{\gamma}_i \lambda_j}}$$
$$q_\gamma = \frac{\sum E^{\gamma}_{(hkl)^{\gamma}_i \lambda_j}}{\sum E^{\alpha}_{(hkl)^{\alpha}_i \lambda_j} + \sum E^{\gamma}_{(hkl)^{\gamma}_i \lambda_j}} \quad (2)$$

where $E^{\alpha}_{(hkl)^{\alpha}_i \lambda_j}$ and $E^{\gamma}_{(hkl)^{\gamma}_i \lambda_j}$ are respectively the total diffracted energy from the phases α and γ of diffraction planes $(hkl)^{\alpha}_j$ and $(hkl)^{\gamma}_j$ for wave length λ_j, denoted as E^{α}_{ij} and E^{γ}_{ij}. E^{α}_{ij} is calculated from the integral intensity at a diffraction peak *(hkl)* as

$$E^{\alpha}_{\mathrm{ij}} = \sum_{k=1}^{n} x_k z_i \quad (3)$$

where $x_k = 2(\Theta_i - \Theta_{i-1})$ is the step angle and y_i is the X-ray count at diffraction position $2\Theta_i$. Table III is the measurement conditions for doublex austenite-ferrite stainless steel SCS14.

TABLE III
CONDITIONS FOR PHASE QUANTITATIVE MEASUREMENT

Measurement method	Fixed-η Ω type
X-ray characteristic	Cu- K_α, λ = 1,54 A°
Filter	Ni foil
Preset time	5 seconds
Step size	0.02°
Voltage and current	20 kV, 10 A

Table IV compares the phase compositions of SCS14 determined from (2) and the microscope imaging method. The strong agreement between the two methods shows the validity of (2) and the computation software can be used effectively in the industrial production.

TABLE IV
PHASE COMPONENTS OF STAINLESS STEEL SCS14

Methods \ Phase	Austenite	Ferrite
X-Pro 1.0	55 %	42.2 %
Microscope Imaging	55.9 %	42.9 %

F. Hardness Determination

Many evidences show that the hardness of crystalline materials has relation to the broadness of the diffraction line. A previous research has determined a linear relation between the Rockwell hardness HRC and the half-width B of the diffraction line of quenched and tempered carbon steel [8]. XPro v1.0 is integrated with function for determining the hardness of quenched carbon steel experimentally. In the case of the Gaussian curve method,

$$\mathrm{HRC} = 87.85B + 20.34$$

where $B = 2\sqrt{2\ln 2}\sigma$ is the half-width of the diffraction line and σ is the standard deviation of the Gaussian curve.

In the case of the parabola method,

$$HRC = 103.01B + 21.31$$

where B is the full width at half of maximum X-ray intensity of the parabola fitted to the diffraction line.

Table V is the measurement conditions for eleven quenched and tempered specimens in water for 45 minutes. Fig. 7 is the relation between the HRC hardness and the line half-width. The straight lines in Fig. 7 show that the developed XPro 1.0 can determine the hardness from the X-ray diffraction measurement data.

TABLE V
CONDITIONS FOR HARDNESS MEASUREMENT OF QUENCHED CARBON STEEL

Measurement method	Fixed-η Ω type
Diffraction plane	(211) of body-centered cubic ferrite
Bragg angle	82.3º
X-ray characteristic	Cu- K_α, λ = 1,54 A°
Filter	Ni foil
Preset time	5 seconds
Step size	0.04º
Voltage and current	20 kV, 10 A

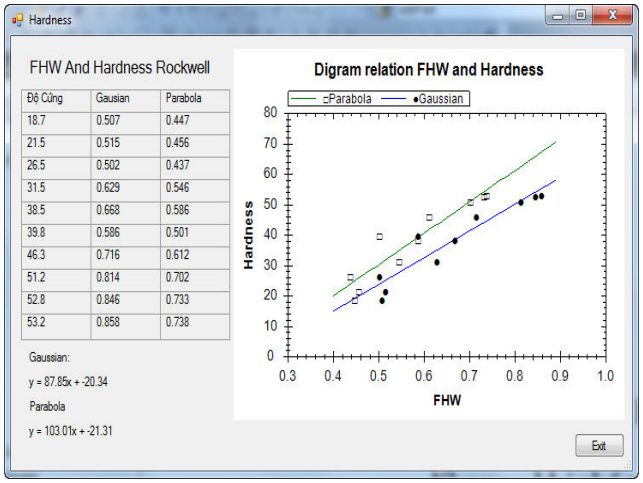

Fig. 7 Dialog box for hardness determination

III. CONCLUSIONS

A new material analyzing software X-Pro 1.0 has been developed with various computational functions to evaluate material properties such as residual stress, hardness, phase quantity; the calculation for different measured data has shown that the computation has very high accuracy.

Further development of the research could be:

a. Development of phase quantitative analyzing for material having three phases or more;
b. Development of hardness of many advanced alloy materials such as nikel-based alloy and aluminum alloys;
c. Determining the thickness for coating layers
d. Integration of function of corrosion mapping using ultrasonic technique;

REFERENCES

[1] V. Hauk, *Structural and Residual Stress Analysis by Nondestructive Method*, Elsevier, 1997,pp 136-168
[2] Lu, J. et al., (1996), *Handbook of Measurement of Residual Stress*, Society for Experimental Mechanics, The Fairmont Press Inc., 225-231.
[3] Noyan I. C. and Cohen J. B.; *Residual Stress Measurement by Diffraction and Interpretation*, New York: Springer-Verlag; 1987.
[4] B. D. Culity, *Element of X – Ray Diffraction*, Prentice Hall Upper Ssddle River, 2001, pp. 256-270.
[5] Le Chi Cuong, *Development of Automated X – Ray Stress Measurement with Its Application*, Doctoral Thesis, Nagaoka UT, Japan, 2004.
[6] Lê Chí Cương and Lê Hoàng Anh, *Structural Analysis for Nano Ceria Using Fourier Analysis for X-Ray Diffraction Line*, The 2012 International Conference on Green Technology and Sustainable Development , 2012, pp.255-259.
[7] Lê Chí Cương and Văn Quốc Hữu, Xác định tỷ lệ pha của thép không gỉ song pha Ferrit và Austenite có độ bền cao bằng nhiễu xạ x-quang, Luận văn thạc sĩ ĐH Sư Phạm Kỹ Thuật TP.HCM, 2011.
[8] Le Chi Cuong, Determination of Hardness Of Quenched And Tempered Steel Using X-Ray Diffraction, Journal of Vietnam Mechanics, No. 4, 2013, pp.54-58.
[9] Andrew Hind, The determination of thin film thickness using reflectance spectroscopy, Agilent Technology, Mar. 2011.
[10] Le Chi Cuong, *Development of a New Automated X-Ray Stress Analyzer*, The Japanese Society for Non-Destructive Inspection, No. 21-25, 1999, pp. 389-394.
[11] Trần Đình Huế, Nguyễn Mạnh Hùng, *Orientation Programming*, 2013, (to be published).
[12] Erik Brown, *Windows Forms Programming with C#*, Manning Publications Co., 2002.
[13] Ian Griffiths, Matthew Adams, Jesse Liberty, *Programming C# 4.0*, O'Reilly, 2010.
[14] L.C. Cương and M. Kurita, *Absorption Factor and Influence of LPA Factor on Stress and Diffraction Line Width in X-Ray Stress Measurement With and Without Restriction of X-Ray Diffraction Area*, The Japanese Society for Experimental Mechanics, 2004, pp.37-43.
[15] Lê Chí Cương and Nguyễn Thị Kim Uyên, *Residual Stress Determination of Friction Stir Welding of 1060 Aluminum Alloy Using X-Ray Diffraction*, Journal of Technical Education, UTE-HCMC, No. 25, 2013.

Asso. Prof. Dr. Le Chi Cuong graduated the Master and PhD. Courses from the Nagaoka University of Technology (Japan) in 2005. He is the member of ASNT, JSNT, and ASME. His research areas include mechanical engineering system and material science. The research topics are automation, and material property analyzing using nondestructive techniques.

After graduating he had three years working in the steel company. November 2013, he was recognised by The Vietnamese Ministry of Education and Training as Associate Professor. Currently, he is the Head of the Automation Technology Department, University of Technical Education-HCMC, Vietnam. His current interests may be listed as X-ray analysis, NDT inspection, and welding automation.