Effect of Mylar film on X-ray stresss measurement of the specimen

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Abstract

The residual stress of α-Fe and beryllium specimen surface with and without the Mylar film are measured by X2001 stress analyzer. The reason and the regulation of the measured residual stress change are discussed. The results show that the increasement of the calibration distance D for the specimen surface with the Mylar film shifts the diffraction peak to the lower angle, thus changing the measured value of residual stress. The real residual stress of specimen can be obtained by correcting the measured value of residual stress.

1 Introduction

There exists a great deal materials with very strong chemical toxicity in nuclear industry, where residual stress must be produced in the machining process. It have been verified that residual stress will make some important effects on use, so it is necessary to measure residual stress in specimen experimentally. However, beacause of very strong chemical toxicity for the material, the experimenter must be poisoned and the instrument must be polluted if a conventional stress measurement method is adopted. Therefore, it is need to find a special stress measurement method.

Residual stress in a polycrstal can be nondestructively measured by X-ray diffraction method, which has been use extensively in industry and laboratory. P.L.Wallance et have been use mylar film when they analyzer phase composition of a poisonous material by X-ray diffraction method, so residual stress in poisonous material may be measured by proofing the poisonous material with mylar film. The stress measurement of specimen with mylar film by X-ray stress measurement has been developed. The results show that X-ray can penetrate through the mylar film and the change of diffraction intensity is very less, but the stress measured value exists some difference between the specimen with and without mylay film. The reason and regulation of stress change for the specimen with mylar film have been studied experimentally in this paper, and a revise method has also been acquired.

2 Experiment procedure

2.1 Experiment method

Resdiual stress is measured using X2001 stress analyzer, which a new modified ψ goniometer is adopted, as shown in Fig[1]. The incident X-rays is normal to the specimen surface and two position detectors A and B symmetry on the incident X-rays allow a simultaneous detection of two reflecting lattice planes. This arrangement reduces data collection time considerably and increases the precision of X-ray stress analysis. The specimens are α-Fe and beryllium. Firstly, Residual stres ly no mylar films are coating on the surface of specimen. Next, the mylar film with thickness 0.1 mm is coating on the surface of specimen, then the residual stress at the same position have been measured. As a result, the stress difference at a same point with and without mylar film can be obtained in the X-ray stress measurement.

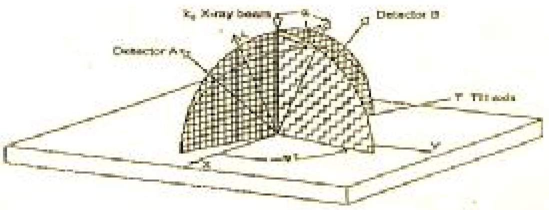

Fig.1 Scheme figure of Modified y-diffraction

2.2 X-ray stress measurement

For α-Fe, X-ray tube is iron tube, the diameter of collimator is 2 mm, the measurement is calibrated using a α-Fe free stress standard specimen. The diffraction angle $2q$ is 145.81°, multiple exposure mode is selected, the tilt angles are 0, ±24.09°, ±35.26° and ±45°, exposure time is 12 sec. The X-ray voltage is 30 kV and the X-ray currents is 5.8 mA. The calibration distance D is 49.49 mm.

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For beryllium, X-ray tube is iron tube, the diameter of collimator is 2 mm, the measurement is calibrated using a Ni free stress standard specimen. Be(103) plane is selected as the diffraction plane, the diffraction angle $2q$ is 145.81°. Multiple exposure mode is selected, the tilt angles are 0, ±24.09°, ±35.26° and ±45°, exposure time is 12 sec. The X-ray voltage is 30 kV and the X-ray currents is 5.8 mA. The calibration distance *D* is 49.30 mm.

3 Experiment Results

The measured stress values of α-Fe and beryllium with and without mylar film are listed in table 1. From the table it is shown that the compressive stress decrease and the tense stress increase when the myalr film is coating on the surface of specimen. The stress difference value between two method is dependent on material property. For α-Fe and beryllium, the average stress difference values are 18.9 MPa and 44.9 MPa, respectively.

Table 1 Residual stress for α-Fe and beryllium specimen with and without Mylar film

Specimen	pt#	without Mylar film σ_1/MPa	with Mylar film σ_2/MPa	$\Delta\sigma = \sigma_1 - \sigma_2$/MPa
α-Fe	1	-258.9	-243.2	15.7
	2	-210.6	-192.2	18.4
	3	164.4	183.6	19.2
	4	219.1	241.3	22.2
Be	1	-193.8	-135.8	58.0
	2	-183.3	-165.7	17.6
	3	-122.8	-74.7	48.1
	4	-98.8	-70.6	28.2
	5	-60.0	-3.2	56.8
	6	-18.9	12.3	31.2
	7	37.9	86.6	48.7
	8	58.7	108.4	49.7
	9	119.6	185.0	65.4

After coating mylar film on the surface of specimen, the diffraction angle at every tilt angle y has also been altered. Taken the measured stress of α-Fe and beryllium in Table 1 as examples, the diffraction angles in stress measurement with and without mylar film are listed in table 2, where $2q_\psi$ is the average value of $2q$ at $+y$ and $-y$ angle. From Table 2, it is indicted that the diffraction angle shifts lower in stress measurement when the mylar film is coating on the surface of specimen, and the shift of the diffraction angle is more evidence when the tilt angle y increase. As a result, the slope of the curve $2q_\psi(\sin^2 y)$ for the specimen with mylar film will change, thus altering measured stress value.

Table 2 Variations of the diffraction angle for both specimen with and without Mylar film

Specimen	Exper.status	$2\theta_\psi$/°			
		$\sin^2\psi=0$	$\sin^2\psi=0.1666$	$\sin^2\psi=0.3333$	$\sin^2\psi=0.5$
α-Fe	without Mylar film	146.14	146.27536	146.31574	146.43711
	with Mylar film	146.08	146.20225	146.26126	146.33115
Be	without Mylar film	142.32	142.35835	142.36713	142.38271
	with Mylar film	142.27	142.30335	142.30988	142.30770

4 Disscusion

The procedure of X-ray stress measurement using X2001 stress anlyzer is as follows: Firstly, the collometer tip contact with the measured point, then taking the contact point as reference location, the gonoimeter is raised to the height, which equals to the calibration distance *D*. When the mylar film is coated on the surface of specimen, the collometer can directly contact with the mylar film, and the reference location is on the surface of mylar film, not on the surface of specimen. Due to the thickness of the mylar film, the height from the mylar film increase, and as is the calibration distance *D*. Just since the calibration distance *D* increase, the measured stress value change when the mylar film is coated on the surface of specimen. Additionally, the mylar film deformed when the collometer contact with the mylar film, so the change of the measured stress value becomes very complication.

4.1 Effect of thickness of mylar film on diffraction angle

As before mentioned, a new modified y goniometer is adopted for X2001 stress analyzer. According to the geometry relations of modified y goniometer, the path changes of diffracted X-ray beams on the surface of specimen with and without mylar film can be obtained. As shown in Fig.2, I_0 is the incident X-ray beams, and the point O is the reference point. If there have no mylar films on the surface of specimen, the point O is located on the surface of specimen, then the diffraction peak is located at point A, and the diffraction angle $2q$ is the angle O_1OA. However, if there have the mylar films on the surface of specimen(which thickness is t), the point O is located on the mylar films, the surface of specimen shifts lower from the point O to the point O_1, and the location of diffraction X-ray beams shifts from the point A to the point B, namely the diffraction angle shifts lower anger, the shift value $\Delta(2q)$ is the angle AOB. From ΔOO_1B in Fig.2, we can obtain:

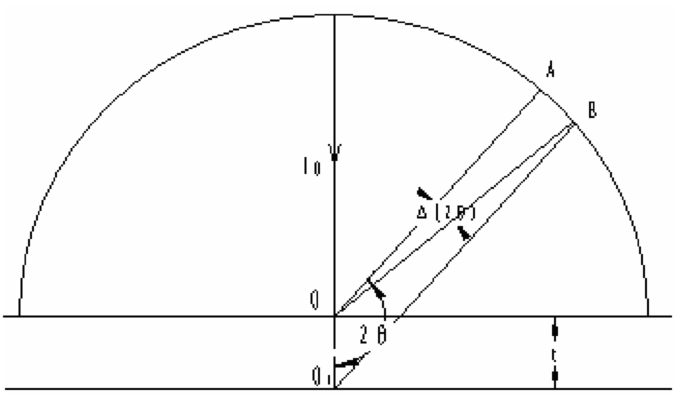

Fig.2 path of X-ray beams with and without Mylar

$$\frac{OB}{\sin(p - 2q)} = \frac{OO_1}{\sin \Delta(2q)} \quad (1)$$

Since OB=D, OO_1=t, t<<D, then

$$\Delta(2q) \approx \sin(\Delta 2q) = \frac{t}{D}\sin 2q \quad (2)$$

Where the unit of $\Delta(2q)$ is arc degree in above eq.(2). If converting arc degree to angle degree, then:

$$\Delta(2q) = \frac{180t}{pD}\sin 2q \quad (3)$$

In fact, the effictive thickness t which incident X-rays beam pentration through mylar film is $t/\cos y$ if the tilt angle of incident X-rays beam increases from 0 to y. As a result, the shift value of diffraction angle is:

$$\Delta(2q_y) = \frac{180t}{pD\cos y}\sin 2q \quad (4)$$

Substituting the experiment parameters of α-Fe and beryllium in this paper into Eq.(4), the shift value $\Delta(2q)$ of diffraction angle can be calculated, which is listed in table 3 for α-Fe and beryllium specimens with and without Mylar film, moreover, the experiment value is also listed in Table 3. The results shows that the shift value $\Delta(2q)$ of diffraction angle by calculation and experiment is basically identical for α-Fe specimen, however, the former is larger than the latter for beryllium specimen. This difference is resulted from two factors: one is the effective thickness decreasing due to the contact between the collometer and mylar film, the other is the statistical error of photon counts from the position detector.

Table 3 The displacement of diffraction angle for both specimens with and without Mylar film

Specimen	The shif value of diffraction angle at every y			
	0^o	24.09^o	35.26^o	45^o
α-Fe	0.06	0.07311	0.05448	0.10595
	(0.06441)	(0.07055)	(0.07888)	(0.09109)
Be	0.05	0.05500	0.05725	0.07501
	(0.07007)	(0.07676)	(0.08520)	(0.09910)

4.2 Effect of myalr film thickness on the measured stress

A modified ψ goniometer is adopted for X2001 stress analyzer, where the stress calculation formula is difference from the conventional X-ray stress analysis:

$$s_f = -\frac{E}{1+n}\cdot\frac{p}{360}\cdot\frac{\cos q}{\sin^3 q}\cdot\frac{\partial(2q)}{\partial \sin^2 y} \quad (5)$$

where E is Young's modulus, n is Poisson's ratio, $2q$ is diffraction angle, y is tilt angle. From Eq.(5), it shows that when the mylar film is coating on the surface of specimen, the variation of the term $\frac{\partial(2q)}{\partial \sin^2 y}$ is resulted in the change of the measured stress. After the myalr film coating on the surface of specimen, the diffraction angle is:

$$2q = 2q_o - \Delta(2q) \qquad (6)$$

where: $2q$ and $2q_o$ are the diffraction angles of the specimen with and without mylar film; $\Delta(2q)$ is the shift value of diffraction angle of the specimen with mylar film, which can be calculated from Eq.(4). Substituting Eq.(6) into Eq.(5):

$$s_f = s_{of} + \Delta s_f \qquad (7)$$

$$\Delta s_f = \frac{E}{1+n} \cdot \cot^2 q \cdot \frac{t}{D} \cdot \frac{\partial(1/\cos y)}{\partial \sin^2 y} \qquad (8)$$

where: $\frac{\partial(1/\cos y)}{\partial \sin^2 y}$ can be calculated from the slope of curve $\sin^2 y \sim 1/\cos y$ by regressive fitting. Under the condition of this paper, $\frac{\partial(1/\cos y)}{\partial \sin^2 y} = 0.82315$. The calibration distance D is about 49 or 50 mm. If D=50mm, substituting them into Eq.(8):

$$\Delta s_f = \frac{0.01646E}{1+n} \cdot t \cdot \cot^2 q \qquad (9)$$

As a result, we can see there exists a linear relation between Δs_ϕ and the thickness of Mylar film. Namely, the more the thickness of Mylar film, the more the difference of the measured stress. Moreover, a linear relation between Δs_ϕ and $\cot^2 q$ also exists, i.e., the larger the diffraction angle $2q$, the less the difference of the measured stress Δs_ϕ. Additionally, the difference of the measured stress Δs_ϕ is dependent on the elastic constant E and ν of the diffraction plane, thus resulting in the difference of the measured stress Δs_ϕ for α-Fe and beryllium.

According to Eq.(9), if no defromation exists when the collometer contacts with mylar film, the calculated Δs_ϕ is 25.29MPa and 60.83MPa for α-Fe and beryllium specimen. However, the measured Δs_ϕ in table 1 is 18.88MPa and 44.86MPa for α-Fe and beryllium specimen. Neglecting the statistical error of photon counts, we can consider this from the contact deformation between the collometer and mylar film, which can be computed as follows:

$$a = \frac{\Delta s_e - \Delta s_t}{\Delta s_t} \qquad (10)$$

where $\Delta\sigma_e$ and $\Delta\sigma_t$ are the stress difference with and without deformation.

From Eq.(10), α is –25.3% and –26.3% for α-Fe and beryllium, which is basically identical. If α is selected –26%, the revised stress formula for α-Fe and beryllium specimen with mylar film can be deduced.

For α-Fe specimen:

$$s_{true} = s_e - 0.74\,\Delta s_t = s_e - 18.71 \qquad (11)$$

For beryllium specimen:

$$s_{true} = s_e - 0.74\,\Delta s_t = s_e - 45.01 \qquad (12)$$

where s_{true} is true stress of the specimen, s_e is the measured stress of the specimen with mylar film.

5 Conclusions

(1) When Mylar film is coating on the surface of the specimen, the diffraction peak shifts lower angle due to the incresement of the calibration distance D, thus incresing the measured value of residual stress. The difference of

the measured stress is direct proportion to the thickness of Mylar film.

(2) When Mylar film is coating on the surface of the specimen, the true stress in the specimen can be calculated from the revised stress formula. For α-Fe specimen, $s_{true} = s_e - 18.71$. For beryllium specimen, $s_{true} = s_e - 45.01$.

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