

In-Situ Strain Measurement on Al7075 Plate by Using High Energy Synchrotron Light Source

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Abstract

In this study, compressive and tensile strain in Al7075 plate have been measured by synchrotron energy-dispersive X-ray diffraction (EDXRD) in conjunction with 4-point bending test under various load up to 1000 lbs(453.6 kg). The compressive and tensile strain are determined by stress related interplanar spacing variation at 111 peak reflection which is considered atomic level strain measurement. The specimen initially shows elastic strain behavior under 400 lbs (181.4 kg) load. However, further load increment applied on sample causes plastic deformation on the regions close edge of the specimen where the biggest stress values in terms of both compression and tension is aroused. Finally, 1000 lbs load is applied on the specimen and major part of the specimen is deformed plastically. Vicinity of body center of sample shows almost no strain formation which is attributed nature of 4-point bending flexural test. Using high energy EDXRD method allow us to reveal precise atomic level strain distribution in Al7075 alloy under different loads.

Keywords: Al7075 alloy, strain measurement, EDXRD method, 4-point flexure test.

Yüksek Enerjili Senkrotron Işık Kaynağı Kullanarak Al7075 Plakalarda Yerinde Gerilme Ölçümü

Öz

Bu çalışmada, enerji dağılımlı x-ışını kırınımı ile Al7075 plakalarda basma ve çekme gerinimi 4 nokta eğilme testiyle 1000 lbs (453.6 kg)'e kadar değişen yüklerle beraber yapılmıştır. Atomik seviye gerinim ölçümü olarak 111 yönünde düzlemlerarası mesafe ile alakalı stres ölçümü ile basma ve çekme gerinimleri belirlenmiştir. İlk olarak numune 400 lbs (181.4 kg) yük altında elastik gerinim göstermiştir. Fakat daha fazla yük artışı basma ve çekme gerinimlerinin ortaya çıktığı yerlerde plastik deformasyona neden olmuştur. Son olarak, 1000 lbs (453.6 kg) yük altında numunenin büyük kısmı plastik olarak deforme olmuştur. Numunenin hacim merkezi civarında 4 nokta eğilme testinin doğası olarak hiçbir strain oluşumu göstermemiştir. EDXRD metodunu kullanmak bize farklı yükler altında Al7075 alaşımında atomik seviye hassasiyette gerinim dağılımını ortaya çıkarmayı sağlamıştır.

Anahtar Kelimeler: Al7075 alaşım, gerinim ölçümü, EDXRD metod, 4 nokta eğilme testi.

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1. Introduction

Four-point bending test method is used to find out flexural strength for several materials system at ambient temperature. Several purposes could be counted for using this test method such as material characterization and development (Mujika, 2006; Quinn & Morrell, 1991). Compressive and tensile stresses are created concomitantly inside of the sample where the sample could be deformed both plastically and elastically (Mujika, 2006; Quinn & Morrell, 1991). The strain measurement with this method is made with micro resolution precision.

EDXRD technique is a powerful non-destructive method for materials characterization which works in transmission (Laue) mode (Tsakalakos, Croft, Jisrawi, Holtz, & Zhong, 2006). So, whole x-ray spectrum could be collected in a very short time period with a stationary Bragg angle (Biçer et al., 2020; Tsakalakos et al., 2006). High energy synchrotron light source could penetrate through even thicker sample so that several characterization type such as lattice strain measurement can be carried out with high spatial resolution in depth of sample (Tsakalakos et al., 2006; Zakharchenko, Gulak, Zhong, Croft, & Tsakalakos, 2003). Interplanar spacing of materials can be directly measured with high precision due to stationary incident and diffracted x-ray (Akdoğan et al., 2012; Şavklıyıldız et al., 2013). Measuring d-spacing variation under several load provides high resolution strain determination with atomic level precision.

There is very limited works presented in the literature about strain measurement with EDXRD method. The main purpose of this study is to reveal a comprehensive atomic level strain measurement with high resolution EDXRD techniques on Al7075 plate specimen which deformed under various load with four point bending test. Understanding strain variation (compressive/tensile) interior of the materials is key to predict materials behavior under different load and improve materials properties for several applications.

2. Material and Method

The experimental work is carried out at the X17-B1 beamline of the National Synchrotron Light Source (NSLS) in the Brookhaven National Laboratory. The EDXRD technique used in this study with photon energy up to 200 keV works in transmission (Laue) mode diffraction where the gauge volume and/or diffraction volume is fixed in space. An illustration about diffraction geometry represented in fig.1 (Akdoğan et al., 2012; Mujika, 2006; Şavklıyıldız et al., 2013; Tsakalakos et al., 2006). Diffraction is conducted by positioning the specimen in x-y-z coordinate system to place the stationary gauge volume into sample. Proper diffraction inside of sample gives suitable peak profiles to measure the peak shifts due to mechanical deformation. It is important that the gauge volume is placed completely inside the specimen because there has to be enough number of grains in the gauge volume for proper counting statistics. The three dimensional size of the gauge volume is adjusted with the slit size and the Bragg angle as illustrated in fig1.

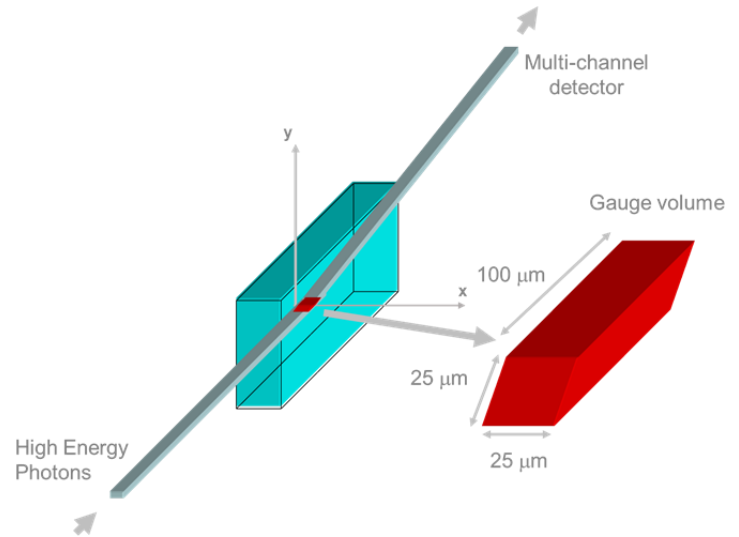


Figure 1. The schematic of experimental setup X17-B1 superconductor wiggler beamline in NSLS showing relative position of the beam path, gauge volume, transmitted beam and diffracted beam detector.

On the other hand, the area exposed high energy x-ray radiation on the specimen surface is perpendicular to the incident beam. The Bragg angle used in this study was kept constant as $2\theta=4^\circ$. Polychromatic radiation is diffracted interior of the sample and the high resolution multichannel Ge detector collect the diffracted radiation.

In this technique, the four point bending test is mounted on an x-y-z stage which is controlled by a micropositioner. Thusly, the gauge volume is first placed on body center of the specimen so as to obtain a diffraction pattern in transmission. Due to the high energy photons, incident beam can penetrate thorough sample and diffract inside the sample. So, this technique make possible to collect data inside of the sample at desired position without cutting or machining the sample. The elastic strain (ϵ_{hkl}) at selected point in the sample is determined by interplanar variation at any (hkl) reflections according to (Tsakalakos et al., 2006);

$$\epsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad (1)$$

where d_{hkl} is the interplanar spacing of selected (hkl) reflection under load and d_{hkl}^0 is the interplanar spacing at zero stress which is measured from as is sample.

Fig. 2 represents four point bending test where the loading span is 1/2 of the support span system is used for Al7075 alloy to measure strain measurement. The distance between inner and outer pins are 10cm and 20 cm, respectively. This 4-point experimental mechanism is placed to the beamline in X17B1 and in-situ EDXRD data collection is conducted under various load. The dimensions of sample is (0.6cm thick x 6 cm width x 30 cm length). Three points are marked as A, B and C on the schematic of the sample. Point B represents exactly body center of the sample which is designated as (0, 0, 0) in x-y-z coordinate system at which no stress formation is detected under various load due to the nature of 4-point test.

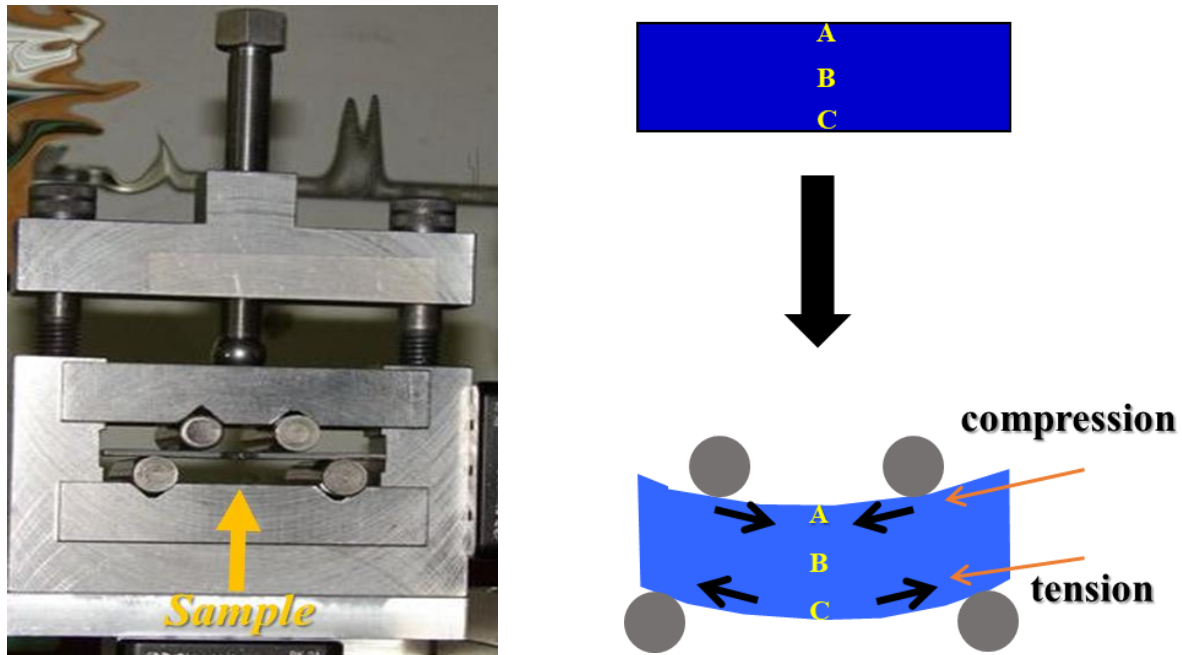


Figure 2. Illustration of 4-point bending experiment and schematic of marked point on sample.

Considering the sample thickness as 6mm, Point A indicates upper edge of the sample (in coordinate designation: (0,3,0)) on which maximum compression stress is aroused under various load. The bottom of the sample is marked as Point C (0, -3, 0) where maximum tension stress is observed under applied load. The EDXRD technique allow to collect very precise data collection which is employed from position (0,3,0) to (0,-3,0).

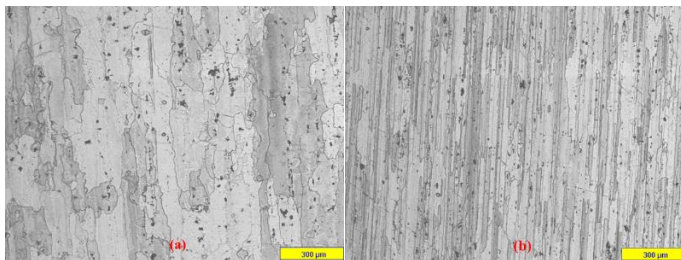


Figure 3. Microstructure of Al7075 Alloy on (a) top view and (b) side view

Considering 150 micron step size, xrd data is collected at 40 different points in 6mm thick sample which suggests that each grain is analyzed through sample loading direction. The Al7075 plate sample with that 4-point test is carried out, is produced by cold rolling process which cause elongation or stretching of grains along the deformation direction. So, the microstructure taken from side view of sample shows elongated grain formation (fig. 3b) where the microstructure at top view of sample reveals equiaxed grain distribution as illustrated in fig. 3a. The microstructures presented in fig. 3 depicts course grain (CG) structure for Al7075 alloy so, this alloy is termed as CG-Al7075.

3. Results and Discussion

Comparison of EDXRD spectra collected different part of sample under 1000 lbs(453.6 kg) load is represented at fig. 4. The data

taken from the most compressed part of sample is represented with black line where the green lines represents most tensed part of sample. The data showed with red line stand for no stress region which is approximately body center of sample. Fig. 4a reveals that no phase transition or new phase formation is occurred after applying load with 4-point test.

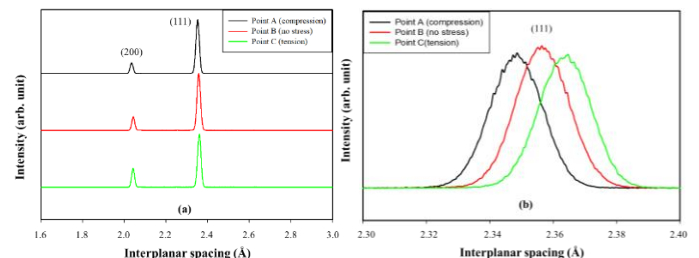


Figure 4. EDXRD data of Al7075 alloy (a) collected at three points and (b) comparison of 111 reflection of all three points when the sample is loaded with 1000 lbs(453.6 kg)

A closer look on the fig 4b suggests that peak shift on the 111 direction of the sample with various stress distribution. When the sample is exposed compression stress, a peak shift on left side which is a decrement on the interplanar spacing so called d-spacing with the compression load. Tensile stress lead increase on the interatomic distance which eventually results peak shift on left side as represented in fig. 4b. During 4-point test, no stress formation is observed in the middle or body center of sample due to mechanism of the test. So, the data collected on point B shows no peak shift. A slight decrease on peak intensities for the point A and C is observed due to various stress formation. Both tensile and compressive stress lead the evolution of the crystallite size or coherently diffracting domain size which represents the mean separation between defects or defect clusters in the (hkl) along which crystalline defects are aroused due to compressive and tensile stress formation in our study. This stress formation lead to

localized structural perturbations such as lattice strain, vacancies and their clusters, dislocations, stacking faults etc. which eventually results a decrement on the peak intensity, beside increasing lattice strain and decreasing crystallite size, along with 111 reflections shifts.

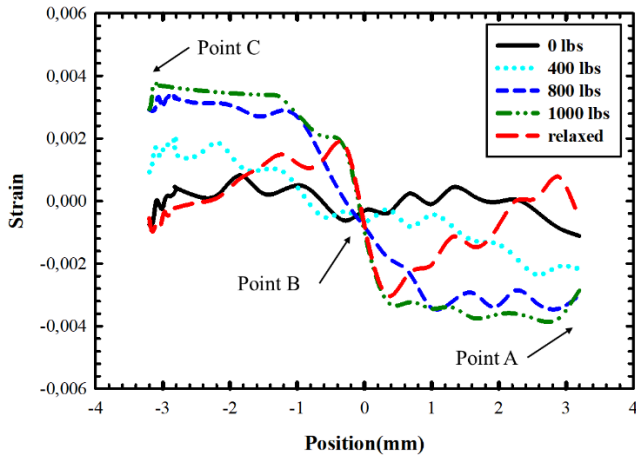


Figure 5. Atomic level lattice strain measurement under various loads.

The lattice strain distribution throughout the sample is represented as function of position under various load in figure 5. EDXRD data collection at each loading is carried out with 150 micron step size from point A to C which creates 40 XRD data in this region. Interplanar spacing of each point detected and strain calculation made concomitantly according to Eq. 1. Gaussian peak fitting is applied on each data and peak positions variation as a function of position is determined. Strain variation between point A to C for as is sample shows almost zero strain line (black curve) which suggests that sample has almost strain free initial structure. When the sample is exposed 400 lbs (181.4 kg), linear strain changes from -0.002 (compression) to 0.002 (tension) is observed between point A-C as illustrated turquoise curve in fig 5. This linear behavior is assigned as elastic deformation in sample. At this point, the sample could return its former shape when the load is released. This much (400 lb) load only stretch the atomic bonding. At Point A, -0.002 strain value is observed which maximum compressive strain. From point A to B, compressive strain decrease down to zero. Due to nature of 4-point testing, there is no stress formation is observed at the exactly body center of sample. From point B to C, the strain variation turn into compressive form up to value of 0.002. When the load is increased 800 lbs (362.8 kg), sample displays two different strain behavior as showed blue curve in fig. 5. Similar linear variation in strain is observed at position from -1mm to 1mm which is elastic strain with the value from -0.0035 to 0.0035. However, outer side of this region, strain variation is almost stabilized which is attributed the plastic strain formation. X-ray diffraction has ability to measure the interplanar spacing or d-spacing which could be interpreted as interatomic distance. When the sample is reached the specific stress for plastically deforming, atomic stretching reaches upper limit and after this point dislocation movements are triggered on specific slip directions. In crystallographic terminology, no more peak shift at peak position is observed beside peak broadening and decrement on peak intensity. This behavior is attributed an increase on dislocation density and a decrease in crystallite size or coherently diffracting domain size due to plastic deformation. In precise version, elastic strain formation is limited in the position between -1mm to 1 mm

of sample where other regions are deformed plastically. Increasing load to 1000 lbs (453.6 kg) deform major part of the sample plastically except the region between -0.2 mm to 0.2 mm as displayed green curve and upper strain value in terms of both tensile and compressive is almost stabilized at 0.0035 and -0.0035, respectively. When the load is released (red curve), stress accumulated during 4-point test keep the elastic region (-0.2mm to 0.2 mm) stable. However, the strain value at both point A and C goes down to zero due to excessive tensile/compressive stress accumulated on both Point A and C. even though there is almost zero strain on both Point A and B, the sample has still deformed plastically except the region between -0.2 mm to 0.2 mm.

4. Conclusions and Recommendations

The results presented in this study reveal a detailed picture of a high resolution synchrotron EDXRD experimental technique along with 4-point bending test which is employed on Al7075 plate to reveal both compressive and tensile strain formation. The sample shows only elastic strain formation under 400 lbs load. However, 800 lbs and 1000 lbs loads yield materials plastically at the most deformed regions. The body center of sample stay non-deformed due to practice of 4-point bending test. Strain measurement on Al7075 plate by using EDXRD method reported in this study are the first in the open literature. The results acquired in this study extend our fundamental knowledge of the mechanical properties of Al7075 and stress response behavior in terms of atomic level under various load. High energy EDXRD strain measurement along with the four point bending test makes this study distinguish and which is first time presented according to open literature.

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