# In-situ Time-resolved Neutron Diffraction Measurements of Thermal Stresses During Severe Thermo-Mechanical Deformation

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### 1. Introduction

The transient materials behavior under the severe thermo-mechanical deformation is one of the most important yet least understood research areas in materials science and engineering. In order to observe the transient materials behavior as it evolves thermomechanical synthesis environment, we adopted a friction stir processing (FSP) system [1].

FSP technology has been growing in interests and aggressively pursuing of applications in industries. The key factor is the temperature and stress distributions, particularly near the rotating tool where the material is stirred and deformation is formed. There have been many studies on this subject both experimental and analytical based [2]. However, direct experimental measurements of the temperature and stress has been extremely challenging, as the severe deformation and phase change associated with the process make many measurement techniques inapplicable.

The deep penetration capability of neutrons into most metallic materials makes neutron diffraction a unique and powerful tool for the investigation of their structures and properties. In this study, we have successfully demonstrated a new measurement method and data analysis approach to determine the transient and dynamic variations of temperature and thermal stresses under severe thermo-mechanical deformation using in-situ time-resolved neutron diffraction.

## 2. Methods and Results

## 2.1 Experimental details

We installed the engineering-scale FSP machine inside the beam room of the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at Los Alamos Neutron Science Center [3]. The in-situ neutron diffraction measurements were performed in a 6.5-mm thick 6061-T6 Al alloy plate during FSP.

For each neutron experiment, a new Al plate was used; the measurement position was predetermined and fixed relative to the tool head. In order to study the evolution of the thermal stresses as the material exits the deformed region, all measurements were made along the centerline and behind the rotating tool. The measurements were made at 5, 8, 10, 15, 20, 30, 50, 70, 100 mm from the rotating tool, as illustrated in Fig. 1(a).

It is noted that three locations (5, 8, and 10 mm) were underneath the rotating shoulder.

#### 2.2 Data analysis methodology

Neutron diffraction is a volume-averaged measurement of interplanar spacing (d-spacing) in a crystalline material based on the Bragg's law from which the apparent lattice strains can be determined. Note that the Rietveld refinement of diffraction patterns for the determination of lattice spacing (d-spacing) was obtained using the General Structure Analysis System (GSAS) [4].

To determine the thermal stresses in the FSP, it was necessary to measure the d-spacings with their scattering vectors parallel to three orthogonal directions of the plate, i.e., longitudinal (x), transverse (y), and normal (z) directions. The neutron scattering volume was  $3 \times 2 \times 2 \text{ mm}^3$ , defined by the 3-mm wide radial collimator of the detector and the  $2 \times 2 \text{ mm}^2$  incident beam square slit. It was centered on the mid-plane of the Al plate as shown in Fig. 1(b).



Fig. 1. (a) Beam position of the in-situ neutron diffraction measurements and (b) Setup of the FSP machine and neutron diffractometer.

## 2.3 Quasi-steady state and changes of d-spacing

Figure 2 shows representative d-spacing changes measured in an experiment. The measurement position in this experiment was located at 15-mm from the rotating tool center. There was a drastic increase in dspacing at the beginning of the experiment occurred approximately for the first 3 minutes (15~18 min of elapsed time). This was associated with the initial transient stage in FSP cycle during which the rotating tool plunges into the material and the build-up of the temperature and stress. After this initial transient, the dspacing remained relatively consistent for about 18 minutes, as labeled in the figure. This confirmed that the quasi-steady state condition was achieved in the experiment [5]. The d-spacing finally decreased after the FSP was completed and the plate began to cool down.



Fig. 2. Evolution of the d-spacing during the neutron experiment at 15 mm position. Note that TD is d-spacing of transverse direction measurement during processing.

## 2.4 Determination of Thermal Stresses

Lattice spacing change for in-situ measurement includes both the elastic strain due to the thermal stress and the thermal strain due to the temperature change. Therefore, it is necessary to separate the thermal strain and the elastic strain from the measured lattice strain. Detail of the data reduction methodology was described in ref. 6.

As shown in Fig. 3, both the longitudinal and transverse stresses were analyzed as a large compression under the tool shoulder. It is due to the thermal expansion and the forging pressure from the tool shoulder. The longitudinal stress was more compressive than the transverse stress, which can be attributed to the elongated temperature field caused by the moving heat source of friction stir tool along the longitudinal direction of the plate. It is worth noting that the compressive longitudinal stress is in the range of the reported yield strength of Al 6061 at 600 K [7].

Outside the tool, the thermal stresses become tensile to balance the thermal contraction of the processed region when it is cools down. In addition, the longitudinal stress becomes higher than the transverse stress when the distance from the tool center is greater than 60 mm. The magnitudes of the tensile stresses (75 MPa for longitudinal stress, and 60 MPa for transverse stress) are quite close to these of the residual stress in other studies [8].



Fig. 3. Thermal stress distribution along the centerline behind the tool as function of distance from the tool center ( $\sigma_x$ : longitudinal stress,  $\sigma_y$ : transverse stress).

## 3. Conclusions

Thermal stresses during severe thermo-mechanical deformation of Al6061-T6 were investigated by means of in-situ neutron diffraction technique. The deep penetration capability of neutrons made it possible for the first time to obtain the thermal stresses during friction stir processing. A data reduction methodology successfully decomposes the thermal stresses from the measured neutron diffraction data.

## REFERENCES

[1] W. M. Thomas, "Friction stir butt welding", International Patent Application PCT/GB92/02203, UK Patent Application 9125978.8, Dec. 1991, US Patent 5 460 317, 1995.

[2] R. S. Mishra, Z. Y. Ma, Friction stir welding and processing, Mater. Sci. Eng. R, Vol. 50, p. 1, 2005.

[3] M. A. M. Bourke, D. C. Dunand, E. Üstundag, SMARTS-A spectrometer for strain measurement in engineering materials, Appl. Phys., Vol. 74, p. S170, 2002.

[4] A. C. Larsen, R. B. Von Dreele, "General Structure Analysis System, GSAS", LAUR 86-748, 2004, Los Alamos National Laboratory.

[5] K. Masubuchi, "Analysis of Welded Structures", New York, Pergamon, 1980.

[6] W. Woo, H. Choo, D. W. Brown, Z. Feng, P. K. Liaw, S. A. David, C. R. Hubbard, M. A. M. Bourke, In situ neutron diffraction measurement of transient temperature and stress fields in a thin plate, Appl. Phys. Lett., Vol. 86, p. 231902, 2005.

[7] Y. S. Touloukian, R. K. Kirby, R. E. Taylor, P. D. Desai, "Thermophysical Properties of Matter, Thermal Expansion, Metallic Elements and Alloys", New York, IFI/Plenum, 1975.
[8] W. Woo, H. Choo, D. W. Brown, Z. Feng, P. K. Liaw, Angular distortion and through-thickness residual stress distribution in the friction-stir processed 6061-T6 aluminum alloy, Mater. Sci. Eng. A, Vol. p. 64, 2006.