

## Quasi-steady state principle and *in-situ* real-time investigation of transient strains in 6061-T6 Al alloy using neutron diffraction

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### Abstract

Neutron diffraction research has been limited to the “static” behavior of materials since the number of collected neutrons is insufficient to reach the adequate neutron counts in rapid changes of material state. In order to achieve the desired precision for the study of the transient material behavior, we propose an *in-situ* neutron-diffraction measurement method based on the quasi-steady state (QSS) phenomenon. The QSS principle was applied for the measurement of transient lattice spacing changes in a 6061-T6 aluminum alloy plate during thermo-mechanical processing.

### 1. Introduction

A number of thermo-mechanical processes have emerged recently and often results in superior mechanical properties of advanced materials [1]. Examples of the thermo-mechanical process include the equal-channel angular extrusion and friction stir welding. Although the thermo-mechanical deformation causes rapid changes of material states (e.g., strain/stress, temperature, and microstructure), the significant issue, i.e., the material state changes, has rarely been reported [2].

The deep penetration capability of neutrons into most metallic materials makes neutron diffraction a powerful tool in understanding their structures and properties [3]. In particular, *in-situ* neutron-diffraction studies have been used to determine the stress evolution, texture changes, thermal expansion, and phase transformation [4,5]. However, the application of neutron diffraction has difficulties in direct real-time measurements of rapidly changing material states due to the relatively inadequate neutron flux. In order to achieve the desired precision, the measurement time and sampling volume are often increased to yield statistically meaningful diffraction data.

In many thermo-mechanical processes a quasi-steady state (QSS) exists when viewed in an alternate coordinate system. For example, in welding the heat source moves relative to the fixed sample, however, when viewed from a coordinate system moving with the heat source, the QSS condition exists. The QSS facilitates investigating the steady state behavior during processing instead of the transient behavior, since it increases the time available to perform the neutron diffraction measurement [6]. Thus, we suggest that the QSS phenomenon is useful for the application of *in-situ* neutron diffraction to investigate the transient behavior.

In this paper, we present: (i) the principle of the QSS and (ii) the transient behavior of strains in (*hkl*) grains of a polycrystalline Al 6061-T6 alloy during thermo-mechanical processing based on the QSS-basis *in-situ* time-resolved neutron diffraction methodology.

### 2. Quasi-Steady State Principle

The measurement of the time-dependent material behavior requires a tracking of material-state evolutions as a function of time in a specific material point. It is generally measured using the *Lagrangian* coordinate ( $x, y, z$ ), which has a observation point of view fixed always to the specific material point ( $P$ ) during processing, Fig. 1(a). Using the fixed point of view, one can

observe the material-state changes (i.e., transient behavior) at  $P$  as a function of time. On the other hand, if the observation point of view moves along with the process simultaneously, Fig. 1(b), the material state does not change as a function of time and consequently achieves the QSS in the *Eulerian* coordinate  $(\omega, \xi, \psi)$ . In the case that the observation point moves at the same speed and along the same direction as the processing, the QSS condition is established [2].

Figures 1(a) and 1(b) are two snap-shots at each instance (i.e.,  $t_1$  and  $t_2$ ). A heat source traverses from  $O_1$  to  $O_2$  along the processing line on a fixed plate with a velocity ( $V$ ) and an observer investigates the evolution of material state as function of time at material point ( $P$ ). Now, two types of coordinate systems, i.e., the *Lagrangian* coordinate  $(x, y, z)$  and the *Eulerian* coordinate  $(\omega, \xi, \psi)$ , are given. At  $t_1$  the material point ( $P$ ) in the *Eulerian* coordinate  $(\omega_{p_1}, \xi_{p_1}, \psi_{p_1})$  is same to the *Lagrangian* coordinate  $(x_p, y_p, z_p)$ , Fig. 1(a). However, at  $t_2$  the material point ( $P$ ) in the *Eulerian* coordinates varies from  $(\omega_{p_1}, \xi_{p_1}, \psi_{p_1})$  to  $(\omega_{p_2}, \xi_{p_2}, \psi_{p_2})$ , Fig. 1(b). Using the principle of moving point source with time, the two coordinate systems can be related with a constant velocity ( $V$ ) at two instances [7]:

$$\omega_p = x_p - V(t_1 - t_2); \quad \xi_p = y_p; \quad \psi_p = z_p \quad (1)$$

Here, denote  $\Phi_p$  as the material state at  $P$ , then the material state of the *Eulerian* coordinate,  $\Phi_p[\omega_p, \xi_p, \psi_p]$ , can be related to that of the *Lagrangian* coordinate,  $\Phi_p[x_p, y_p, z_p]$ , at each instance using Eq. (1):

$$\begin{aligned} \Phi_p[t_1] &= \Phi_p[\omega_{p_1}, \xi_{p_1}, \psi_{p_1}] = \Phi_p[x_p - V(t_0 - t_1), y_p, z_p], \text{ and} \\ \Phi_p[t_2] &= \Phi_p[\omega_{p_2}, \xi_{p_2}, \psi_{p_2}] = \Phi_p[x_p - V(t_1 - t_2), y_p, z_p] \end{aligned} \quad (2)$$

Supposing the initial state of material is homogeneous within the entire sample, the material state measured at  $t_2$  should be the same to the measured at  $t_1$  (i.e.,  $\Phi_p[t_1] = \Phi_p[t_2]$ ) as shown in Eq. (2). Thus, material state measurements using the QSS in the *Eulerian* coordinate can represent the transient behavior of material state in the *Lagrangian* coordinate [6].

To construct the temporal variations of the transient behavior in the material state, a series of experiments should be performed under QSS in the *Eulerian* coordinate system. Importantly sample traverses along the material flow direction ( $-\omega$  direction) and the multi experiments provide data of a specific material state at each observation position in the *Eulerian* coordinate. Consequently, the material state can be presented as a function of distance between the observation position and the processing position ( $\Delta\omega$ ). The time interval,  $\Delta t_i (= t_i - t_{i-1})$ , which is defined as the elapsed time from one observation position to the next, can be determined by the relative distance ( $\Delta\omega_i = \omega_i - \omega_{i-1}$ ) divided by the constant velocity ( $V$ ) of material flow using the time-

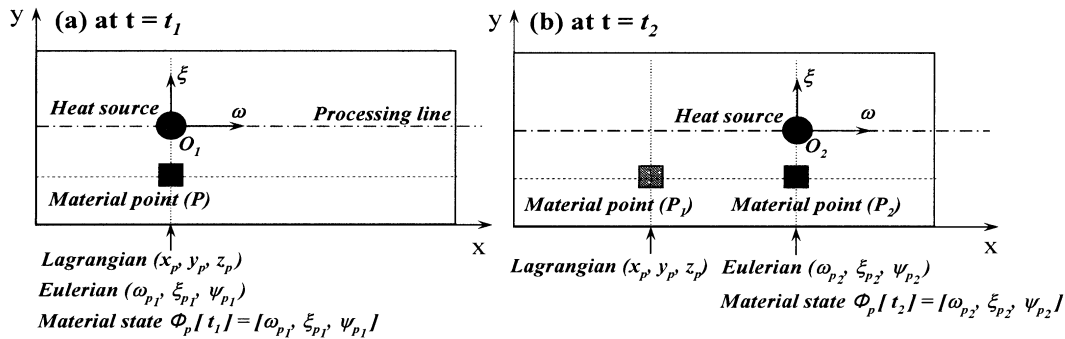


Fig. 1. Principle of the quasi-steady state measurement. *Eulerian* coordinate  $(\omega_p, \xi_p, \psi_p)$  is superimposed on *Lagrangian* coordinate  $(x_p, y_p, z_p)$  at the two instances: (a) before moving of the heat source at time,  $t_1$ , (b) after moving of the heat source from  $O_1$  to  $O_2$  with a velocity ( $V$ ) at time,  $t_2$ . Material states ( $\Phi$ ) were represented at two instances of the material points ( $P_1$  and  $P_2$ )

distance-velocity relationship. Thus, the temporal variation of transient behavior from the each position in the *Eulerian* coordinate can be transformed to the *Lagrangian* coordinate as below:

$$\Phi[x_0, y_0, t_i] = \Phi[\omega_i, \xi_i, \psi_i], \text{ where } t_i = \frac{\Delta\omega_i}{V} + t_0 \quad (3)$$

where  $t_0$  is the reference time when  $\Delta\omega_i = 0$ . Finally, by the conversion from multi QSS measurements in the *Eulerian* coordinate to the *Lagrangian* coordinate, one can obtain the transient behavior of the material state as a function of time.

### 3. *In-situ* real-time neutron-diffraction measurements using QSS principle

Commercial 6061-T6 Al alloy plates were subjected to a thermo-mechanical process, namely the friction stir welding (FSW). Severe plastic deformations were caused by the rotating tool pin and frictional heating was generated mainly from the pressing tool shoulder during FSW [8]. Figure 2 shows *in-situ* real-time neutron experimental setup during FSW. The processing started with the predetermined distance ( $r$ ) between the tool center and scattering volume position, while importantly the whole Al plate traversed at a constant speed. These processing conditions created a uniform material state, i.e. QSS of transient lattice spacing ( $d$ -spacing), during the translation of the Al plate. *In-situ* neutron-diffraction measurements were performed at various positions ( $r = 5$  to 100 mm), Fig. 2, within the deformation path under the constant FSW condition using new plates.

Spatially-resolved time-of-flight neutron diffraction provides  $d$ -spacings, which were diffracted from the grain sets with their reflecting  $hkl$  lattice plane normal parallel to a specific direction of the plate [3]. The diffraction patterns, each scattering vector parallels to the LD ( $x$ ) was measured using

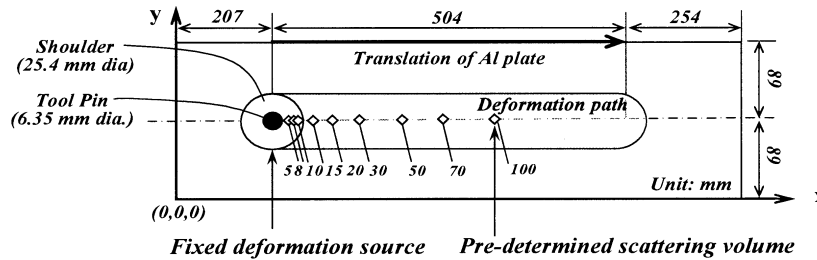


Fig. 2. Experimental setup for *in-situ* neutron diffraction measurements under the friction stir welding. Note that it shows the final position after the translation of the Al plate, and the tool size and deformation path was exaggerated for clarity.

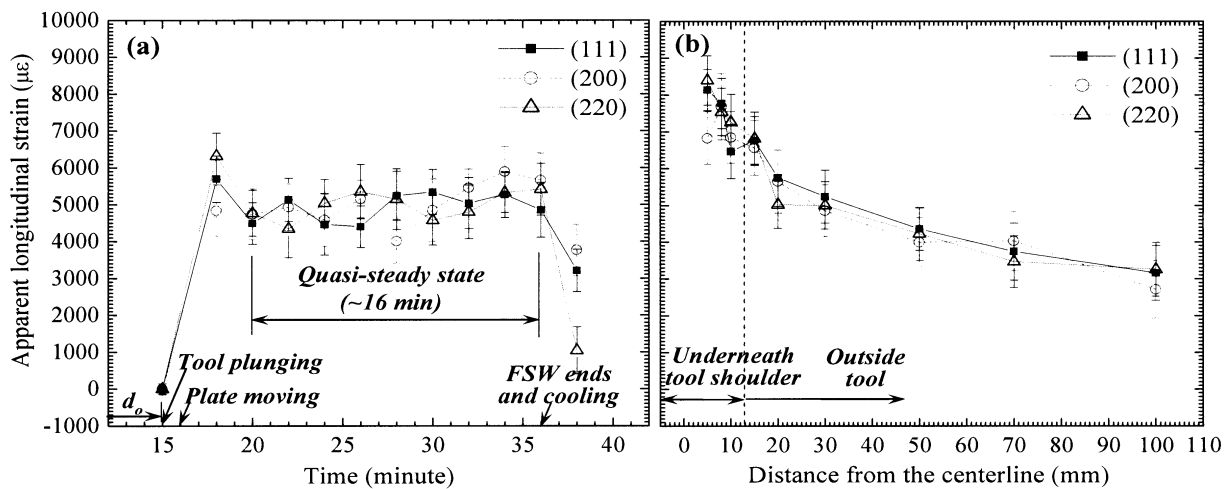


Fig. 3. *In-situ* neutron diffraction results using QSS principle: (a) evolution of the longitudinal strain component during FSW ( $r = 30$  mm). (b) Evolution of strains as a function of distance from the tool center. A total of 9 sets of QSS measurements contribute to construct.

a scattering volume of  $2(x) \times 2(y) \times 3(z)$  mm<sup>3</sup> at the SMARTS (Spectrometer for Materials Research at Temperature and Stress) diffractometer at the Los Alamos Neutron Science Center [9]. Note that the  $d$ -spacing of each  $hkl$  reflections was obtained using the single peak fitting method in GSAS (General Structure Analysis System) [10].

Figure 3(a) shows the lattice strains calculated from the neutron diffraction measurements on a single plate. Initially the reference lattice parameter is measured for 15 minutes while the tool is about 254 mm from one end of the plate. The measurement position for this specific sample is  $r = 30$  mm, i.e. 30 mm behind the tool center. After the  $d_o$  measurement is completed, the tool is plunged into the plate. Once the plate starts translating, a series of 2-minute neutron measurements is started. The translation speed is 0.42 mm/sec allows for about 11 neutron measurements during the 20 minutes of travel time. At the end, the tool has traversed a total of 504 mm along the plate ending up 207 mm from the other end of the plate, see Fig 2. Hence, the LD component of the Al ( $hkl$ )  $d$ -spacing was measured continuously with a 2-minute temporal resolution. The strain ( $\varepsilon$ ) was calculated using:  $\varepsilon = (d - d_o) / d_o$ , where  $d$  is the measured lattice spacing during FSW [3,8]. Fig. 3(b) shows the measured apparent strains ( $\varepsilon$ ) as a function of distance from the tool center constructed using the averaged strains within the QSS period during FSW, Fig. 3(a), for several samples. The measured lattice strains are a combination of the mechanical and thermal strains, and hence the two contributions must be separated before the true transient strains can be determined. A procedure for de-convoluting the mechanical and thermal strains can be found elsewhere [11].

#### 4. Summary

Direct real-time neutron diffraction has difficulties in the measurement of the transient behavior due to the insufficient neutron flux. To explore such transient behavior, *in-situ* time-resolved neutron diffraction measurement based on the quasi-state state (QSS) principle was suggested. Using the QSS-basis measurement method, lattice spacing changes and strains evolution as a function of distance from the deformation were investigated during thermo-mechanical processing.

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