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# Ultrahigh dense and gradient nano-precipitates generated by warm laser shock peening for combination of high strength and ductility



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#### **ABSTRACT**

Nanocrystalline materials generated by severe plastic deformation often come with high strength but low ductility due to the inability to accumulate dislocations and thus the low work hardening rate. In this study, a unique high strain rate deformation process – warm laser shock peening (WLSP) – is studied to generate extremely high-density nano-precipitates in precipitation hardenable alloy. Aluminum alloy (AA) 7075 was selected to evaluate the generation of ultra-high-density precipitates by WLSP and the effects on the strength and ductility. WLSP integrates the advantages of laser shock peening (LSP), dynamic strain aging (DSA) and dynamic precipitation (DP). The nanoscale precipitate particles generated by WLSP effectively block dislocations and thus increase the material strength. The precipitate–dislocation interaction has been observed by high resolution TEM (HR-TEM) and modeled by the multiscale discrete dislocation dynamic (MDDD) model. It has been demonstrated that compared with room temperature LSP, WLSP can improve material strength by 32.3% without compromising the ductility, in that elongation remains 20%. These ultra-high-density nano-precipitates greatly improve dislocation accumulation capacity and thus effectively increase ductility.

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## 1. Introduction

Severe plastic deformation (SPD) has been widely used to improve strength through nanocrystalline formation [1–[4\].](#page--1-0) However, the ductility is often compromised due to the inability for dislocations to accumulate and thus low work hardening rate in nanocrystalline structure. It has been a long time question to find out a microstructure that has high strength without sacrificing the ductility. There have been many attempts to produce materials with both high strength and high ductility [\[5,6\]](#page--1-0), for example, by designing bi-modal microstructures [\[7\]](#page--1-0), gradient nanostructures [\[8,9\],](#page--1-0) by introducing coherent nanoscale twin boundaries [\[10,11\],](#page--1-0) or by precipitate particles [12–[16\]](#page--1-0). Among these methods, precipitation hardening has proved to be an important method to enhance mechanical properties [\[16\].](#page--1-0) The effects of precipitates on property enhancement greatly depend on precipitate size and number density. There have been limited ways to generate ultrafine and high-density precipitates. Recently, generation of nanoprecipitate in aluminum alloy [\[17,18\]](#page--1-0) and carbon steel [\[19,20\]](#page--1-0) has been reported to have promising applications on mechanical properties enhancement, such as fatigue resistance. However, the

<http://dx.doi.org/10.1016/j.msea.2014.05.003> 0921-5093/© 2014 Elsevier B.V. All rights reserved. mechanism of how these nano-precipitates could be used to improve strength and ductility remains unclear. This limits the selection of processing techniques for ideal mechanical properties.

In general, precipitates can effectively strengthen material by imposing resistance to dislocation slip. On the other hand, ductility could also be favorably affected by the precipitates due to the improved dislocation accumulation capacity acquired from dislocation–precipitate interaction. However, in order to take full advantage of precipitates, their size, density and distribution need to be optimized. This study will develop a novel process, warm laser shock peening (WLSP), in order to generate ultrahigh-density nanoprecipitates and significantly increase strength without loss of ductility in precipitation hardenable materials. WLSP integrates the advantages of laser shock peening (LSP), dynamic strain aging (DSA) and dynamic precipitation (DP) [\[17,19,20\]](#page--1-0). In order to make use of WLSP for precipitation hardenable materials with high strength and ductility, there are several critical problems to address. First, what is the technical route for extremely high-density nanoprecipitates and dislocations in metals? Second, how these nanoprecipitates and dislocations interact and thus affecting the strength and ductility of materials? Third, how does the distribution of the nano-precipitates affect the mechanical properties?

In order to address these problems, WLSP of aluminum alloy (AA) 7075 will be carried out in the following aspects. First, the effects of processing conditions of WLSP on dislocation density, precipitate

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density, material hardness, and residual stress magnitude and distribution will be studied. The optical WLSP temperature will be found in order to achieve ultrahigh density of nano-precipitates. Secondly, this study will focus on the dislocation–precipitate interaction and its effects on material strength and ductility. We will investigate the in-depth distribution of the nanoscale precipitates, the interaction between dislocations and precipitates through dislocation dynamics and high-resolution TEM, and how this affects material strength and ductility. This study can further unveil the fundamental mechanisms of WLSP and how it affects the strength and ductility of precipitate-hardenable alloys.

AA 7075 was chosen because it has been widely used in the aerospace industry and its precipitation behavior has been well studied in the literature. There are three kinds of precipitates [\[21\]](#page--1-0) in AA 7075, including coherent, spherical Guinier–Preston (GP) zones with a Zn/Mg atomic ratio of about 1:1; metastable, semicoherent, plate-shaped  $\eta'$  with an atomic ratio of Zn/Mg around 1.5:1; and incoherent, equiaxed, stable hexagonal η precipitates with an atomic ratio of Zn/Mg equal to 2:1. For the coherent GP zones and the semi-coherent  $\eta'$  phase, material strength increases through dislocation shearing; for the incoherent  $\eta$  phase, Orowan strengthening dominates. The precipitation sequence in the Al–Zn–Mg-based alloys can be described as follows: supersaturated solid solution  $\rightarrow$  GP zones  $\rightarrow$  η' phase  $\rightarrow$  η' phase (MgZn<sub>2</sub>) [\[22,23\]](#page--1-0). In this study, the microstructure of AA 7075 after WLSP was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The mechanical properties were evaluated by the hardness test and the tensile test. In addition, the multiscale discrete dislocation dynamic (MDDD) model was used to understand the dislocation evolution as a result of interaction between dislocations and precipitate.

#### 2. Experimental method

#### 2.1. Materials

Rolled AA 7075 sheets (composition in wt%: Zn 5.6%, Mg 2.5%, Cu 1.6%, Fe 0.50%, Si 0.40%, Mn 0.30%, Cr 0.23%, Ti 0.20% and Al balance) purchased from McMaster Carr were used in this study. Before LSP, the samples were solution treated at 500  $\degree$ C for 5 h followed by water quenching, which resulted in a uniform microstructure with an average grain size of around 40  $\mu$ m.

#### 2.2. Laser shock peening

A Surelite III Q-switched Nd-YAG laser (Continuum Inc.), operating at a wavelength of 1064 nm with a pulse width (full width at half maximum (FWHM)) of 5 ns, was used to deliver the pulsed laser. The laser beam size is 1 mm and the laser intensity is 4 GW/cm<sup>2</sup>. The overlap ratio is 75% in both the X and Y directions. Aluminum thin foil (30  $\mu$ m thick) was used as the ablative coating and BK7 glass as the confining media. Both room temperature LSP (RT-LSP) and WLSP were carried out for comparison purpose. During WLSP, the samples were heated to the desired temperatures by a hot plate equipped with a thermocouple.

### 2.3. X-ray diffraction (XRD) phase analysis

XRD was used to characterize the second phase particles by a Bruker D8-Focus system with Cu- $K_{\alpha 1}$  radiation source.

Residual stress measurement was carried out in a Bruker D8-Discover X-ray micro-diffraction system with a Cobalt radiation source. The (331) peak was used for stress analysis, which corresponds to a 2- $\theta$  angle of 148.63 $\degree$  in the unstressed state. The interference lines of the Al phase were determined at 11  $\psi$ -angles

from  $-50^{\circ}$  to  $+50^{\circ}$  using CoK<sub> $\alpha$ 1</sub>-radiation and analyzed by the  $\sin^2 \psi$  method [\[24\]](#page--1-0). The X-ray peak broadenings were evaluated by the FWHM integral values after removal of the  $K_{\alpha2}$  signal. The FWHM values at  $90^{\circ}$  X-ray incidence angle of the Bragg diffraction (400) peaks were used as a measure of their relative dislocation density.

The micro-hardness test was carried out with a Leco M-400-H micro-hardness tester with a 200 g load and a 10 s holding time. An average of five measurements was reported for each data point.

#### 2.4. Tensile test

Samples (thickness 1.0 mm, gauge area: 8 mm by 6 mm) were cut by a water jet cutter from a sheet sample. Both sides of the gauge area are peened before the tensile test. The tensile test was carried out at room temperature with a strain rate of  $5 \times 10^{-4}$  s<sup>-1</sup>. For each condition, five specimens were used to ensure reliability.

#### 2.5. Transmission electron microscopy (TEM)

The TEM samples were prepared by the Focused Ion Beam (FIB) lift-out method in an FEI Nova-200 FIB system at Birck Nanotechnology Center at Purdue University. TEM observations of the precipitates and the dislocations were carried out using an FEI Titan TEM operated at 300 kV. The volume fraction of the precipitates was estimated by Image-J, a professional pixel analyzing software. A relatively large area (5  $\mu$ m by 5  $\mu$ m) of the sample was analyzed to ensure its statistical accuracy.

## 3. Results and discussion

#### 3.1. Optimal temperature for WLSP

After WLSP, material strength is affected by dislocation density and distribution, size and density of precipitates. The nucleation and growth of nano-precipitation, and their interaction with dislocation during LSP at various temperatures varies for different material systems [\[19\].](#page--1-0) Therefore, the optimal temperatures for WLSP processing are material dependent. In order to find out the optimal temperature for WLSP processing of AA 7075, WLSP was carried out from 100 °C to 300 °C with a 50 °C interval. As shown in Fig. 1, material hardness increases gradually as temperature increases from 100 °C to 250 °C. When the temperature reaches 300 °C, there is a significant hardness drop. It can be concluded that  $250 \degree C$  is the optimal temperature for WLSP of AA 7075 from the perspective of precipitate strengthening. This temperature will be used in this work to generate nano-precipitation and high dense dislocations, and study the microstructure changes such as dislocation density



Fig. 1. Hardness as a function of WLSP temperature.

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