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Photoacoustic Studies of Nanocrystalline Ag and Al$_2$O$_3$

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Abstract. — The photoacoustic frequency spectrums of nanocrystalline Ag and Al$_2$O$_3$ versus the grain size and the annealing temperature respectively were investigated by using synchrotron radiation X-ray photoacoustic experiments.

1. Introduction

Nanocrystalline materials have also been called ultra-fine grained materials, nanophase materials or nanometer-sized crystalline materials [1]. In the case of nanocrystalline materials, the generation of solids with new atomic structures and properties was attempted by using the atomic arrangements in the cores of defects such as grain boundaries, interphase boundaries or dislocations. Since the atomic structure of nanocrystalline materials differs from the structure of glasses and crystals, the structure-dependent properties of the chemically identical substances in the glassy or crystalline state, therefore, photoacoustic effect from nanocrystalline materials may exhibit attractive different features. As we know, the photoacoustic effect has become a subject of intensive research because of its fundamental significance and potential for applications. However, until recently, the photoacoustic technique has been confined in the infrared, visible and ultraviolet regions of the spectrum since the intensity of conventional light source in the higher energy region is too weak to induce detectable photoacoustic signal. With the advent of high brilliance synchrotron radiation, it is possible to extend photoacoustic technique to the X-ray region of the spectrum. So far photoacoustic effect induced by X-ray absorption from nanocrystalline is still a relative new subject, little attention is paid to the mechanisms of heat production and transfer in nanocrystalline. This is the purpose of our experiments. In this paper the photoacoustic frequency spectrums of nanocrystalline Ag and Al$_2$O$_3$ as a function of the grain size and the annealing temperature respectively was investigated by using synchrotron radiation X-ray photoacoustic experiments.

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The Sketch of Photoacoustic Cell

Fig. 1. — The sketch of the Photoacoustic cell.

2. Experiments

The experimental arrangement is the same as that shown in the literature [2,3]. The sketch of the photoacoustic cell for multichromatic synchrotron radiation X-ray experiments is shown in Figure 1, which was designed for X-ray absorption studies of solid samples, and is similar to the one described in reference [4]. The brass cell has a volume of 18 mm dam. × 5 mm, the Beryllium foil (18 mm dam. × 0.2 mm thickness) was used for the cell window. The microphone was a very cheap and commercially available electret condenser type (0.7 mm dam., sensitivity 32 mV/Pa). The chamber was connected to the microphone by a small channel (1 mm dam. × 7 mm thickness). Samples to be studied were adhered to the inner side of exit Beryllium window.

The experimental set-up for X-ray photoacoustic effect is shown in Figure 2. X-ray were chopped by a rotating blade made of Al plate (3 mm thickness), and X-ray photon flux was monitored by an ion chamber (thickness of Ar gas 2 cm). The photoacoustic signal was fed to an EG&G PARC model 124 lock-in amplifier. The experiments were performed on the wiggler photon beam line 4W1A (a multichromatic X-ray source, 0.4 - 3 Å) at our laboratory.

The bulk nanocrystalline Ag materials were prepared by the inert gas condensation and in situ compacting technique. The two samples of nanocrystalline Ag have the same diameter 6 mm and thickness 140 μm with different crystal size 17 nm and 33 nm respectively, which were studied by TEM (Transmission Electron Microscopy). The nanocrystalline Al₂O₃ clusters were generated by the chemical reaction method and the bulk ceramics Al₂O₃ were formed by compacting. The three samples of nanocrystalline Al₂O₃ have the same diameter 12 mm, thickness 1.5 mm, compacting pressure 150 MPa, and annealing time 4 hours with different annealing temperature 100 °C, 700 °C and 1280 °C, respectively.

3. Results and Discuss

The experimental results were shown in Figures 3 and 4. PAS is the amplitude of photoacoustic signal, f is the chopping frequency. The amplitude of photoacoustic signal of nanocrystalline Ag material with grain size 33 nm is more great than that with 17 nm. The amplitude of
The Experimental Setup

SR X-ray \rightarrow Silt \rightarrow Ion Chamber \rightarrow Photoacoustic Cell

BEPC Storage Ring

Storage Oscilloscope \rightarrow Lock-In Amplifier

Fig. 2. — The experimental set-up for X-ray photoacoustic effect.

PAS versus f

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{fig3}
\caption{The amplitude of photoacoustic signal (PAS) of nanocrystalline Ag as a function of the chopping frequency $f$.}
\end{figure}

The photoacoustic signal of nanocrystalline Al$_2$O$_3$ increases as the annealing temperature raises. It is the basic idea of nanocrystalline materials to generate a new class of disordered solid by introducing a high density of grain boundaries. Nanocrystalline metals exhibit crystal growth at elevated temperatures. Similar grain growth effects have been noticed in nanocrystalline ceramics. The density of grain boundaries decreases with grain growth. The photoacoustic response to X-ray absorption results from the thermalization of the excited Auger electrons and photoelectrons. The excited electrons have a small mean free path 10-100 Å (smaller than the grain size) and a de Broglie wave length 1-10 Å (much smaller than the grain size). Whereas we may estimate that the heat production from nanocrystalline may not change much with the
Fig. 4. — The amplitude of photoacoustic signal (PAS) of nanocrystalline Al₂O₃ as a function of the chopping frequency f.

In summary, the photoacoustic frequency spectrums of nanocrystalline Ag and Al₂O₃ versus the grain size and the annealing temperature respectively were studied by using synchrotron radiation X-ray photoacoustic experiments. The production of heat flow resulting from X-ray absorption in the sample is quite different from that resulting from laser absorption. The energy of the X-ray photons is much higher in comparison with the photons from conventional light sources (laser, xenon lamp, etc.), therefore, for X-ray absorption, heat is mainly generated through excitation and de-excitation of inner shell or core electrons instead of outer shell or band-gap low-energy electrons. The heat transfer in nanocrystalline Ag and Al₂O₃ is related to the grain size. The thermal wave was intensely scattered by the grain boundaries. The photoacoustic effect of nanocrystalline induced by X-ray absorption is still a new field. The heat production mechanisms and diffusion processes are needed to be further investigated for nanocrystalline with a high density of grain boundaries.
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