3Pb1-3

Acoustic properties of metal close to the melting point measured by laser ultrasonics
レーザー超音波法で測定した融点近くの金属の音響物性

Hisato Ogiso[1], Harumichi Sato[1], and Hirotomo Itagaki[1] ([1] AIST)
小木曽久人[1], 佐藤治道[1], 板垣宏知[1], ([1]産総研)

1. Introduction

Acoustic properties at high temperatures and high pressures （HTHP） have widely attracted attention. Especially these properties are crucial for the geophysical study of the Earth's mantle and core, because seismic waves have been employed to examine the internal structure of the Earth. Under this background, a number of studies were conducted to measure the acoustic properties at HTHP.

Some of these studies employed the methods based on phone-photon interactions to indirectly obtain the sound velocities. There were however discrepancies among the velocities obtained with different methods. Whereas laser ultrasonics (LU), as a direct measurement method, appears to be one of the most promising methods at high temperatures. Using LU, Zinin et al. obtained the iron's sound velocity of both longitudinal and shear waves at 22 GPa and 2590 K[1]. This result proves the effectiveness of LU on examinations of acoustic properties at HTHP.

In this study, we applied LU to examine the acoustic properties at the temperature closed to the melting point. The preceding studies were conducted at the stable solid states, whereas this study has been focused on the neighbor of the phase transition. Lead-free solders, as an alternative lead solders, still have problems in which the formation of inter-metallic compounds causes the deterioration of the joint strength[2]. To clarify the solder's behaviors on the acoustic properties closed to the melting point should help to solve the problems.

2. Methods

The schematic view of the laser ultrasonics with sample heating system is shown in **Fig.1**. The whole system is composed of the laser ultrasonics system and the temperature control system.

2.1 *Laser ultrasonics system*

The nano-pulse laser with a pulse width of 5 ns and a wavelength of 527 nm was used for acoustic excitation. The galvano scanner two-dimensionally positioned the laser beam on the sample surface, at which the laser was focused with the spot size of no more than 100 µm using a beam expander and an objective lens. The repetition of the laser pulse was 1 kHz, and the laser instrument

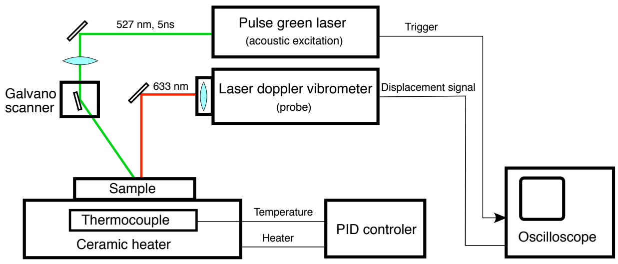

Fig. 1 Experimental setup of the measurement system of laser ultrasonics with elevating sample temperatures.

outputted the trigger signal to synchronize the data acquisitions with the laser pulse outputs.

The laser doppler vibrometer was used for the LU signal, detecting the displacement of the surface position at which the probe laser (633 nm) was irradiated.

2.2 *Temperature control system*

A ceramic heater panel was employed to heat a sample, the heater was permitted the temperature to rise up to 500°C. A K-type thermocouple sensor was buried inside the heater panel and measured the temperature. A PID controller was connected with the heater and the thermo couple, then controlling the temperature to keep it the set-point temperature. The PID controller was provided the temperature to be set within 0.1°C resolution.

2.3 *Sample preparation*

First, we prepared an oxygen free copper substrate (JIS:C1020) with the dimensions of $40 \times 40 \times 5$ mm^3, and a lead-free solder wire, the solder was made of Sn-3Ag-0.5Cu, the nominal solidus temperature is 217°C and the nominal liquidus temperature is 219°C.

We engraved a round-shape hollow with the depth of 3 mm on the copper substrate, then spirally inserting the solder wire to the hollow.

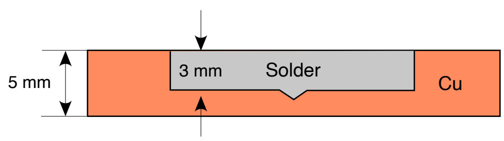

Fig. 2 Schematic profile view of solder sample, the small indentation at the center is fabricated by a center guide tool.

Subsequently, we heated the substrate up to 300 °C, resulting in that the hollow was filled with the liquid solder. After the substrates was cool down to the room temperature and the solder was solidified, we polished the surface of the substrate to flatten the surface and to remove the wax ingredient extracted from liquid solder. **Fig. 2** shows the sample profile.

2.4 *Measurement procedures*

We positioned the excitation laser using the galvano scanner, setting the distance from the probe laser to be 1.52 mm. This distance was fixed during the laser ultrasonic measurements. As varying the temperature of sample, the waveform was recorded. First, we set the temperature from 40°C up to 200°C with an interval of 40°C, then from 200°C up to 220°C with an interval of 1°C.

3. Results

Fig. 3 shows the waveforms at the representative temperatures. Three types of the waveforms can be seen, first one, hereafter named type A, is from 40°C to 202°C, second one hereafter named type B, is from 203°C to 208°C, third one, not having meaningful waveforms, is at 209°C and higher.

Type A is seen in wide range temperature, and the waveform change in the type A is gradual, which is delayed and is diminished with increase of the temperature. Whereas the transition from type A to Type B is so sudden, being caused in one degree. Similarly, the undetectable phenomenon suddenly occurred with the rise of one degree from 208°C. These sudden changes of the waveforms suggest that certain kinds of phase transformations are possibly occurred from the view point of acoustic properties.

The velocity of the SAW at each temperature was estimated. The propagation distance was regarded as the distance between the probe and the excitation laser. To evaluate the propagation time, a continuous wavelet transformation applied to the waveforms then extracted the waveforms with the center wavelength of 1.3 MHz. The peak time of the 1.3 MHz wave was regarded as the propagation time. **Fig. 4** shows the velocity at each temperature. In type A region, the velocity gradually decreases with increasing the temperature and the curve looks smooth, whereas, in type B, the decrease of velocity suddenly gets steep. The change of velocity has discontinuity at the boundary between type A and type B. From the point of the velocity, a phase transformation in the lead-free solder should occur from type A to type B.

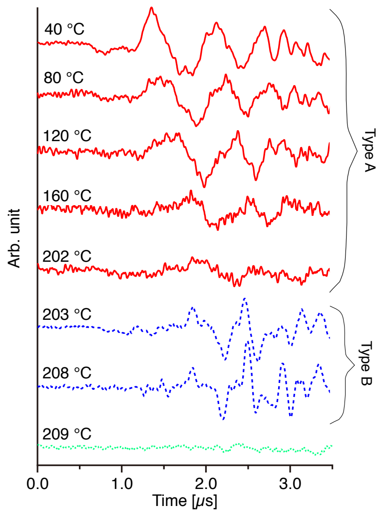

Fig. 3 Waveforms at the representative temperatures, where time = 0 denotes the time at the laser-pulse peak.

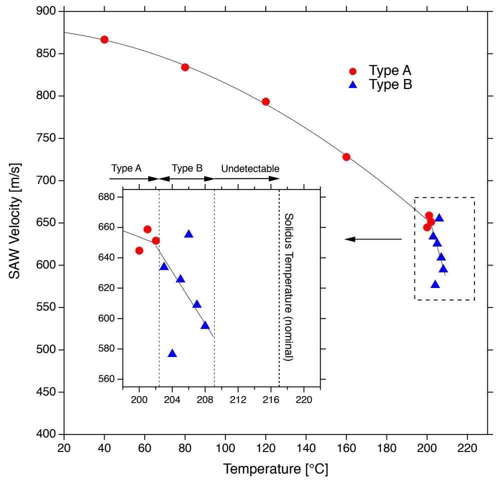

Fig. 4 Relations of the sound velocity to the solder temperature, where ● is type A waveform, ▲ is type B waveform.

The undetectable phenomenon at 209°C and higher also occurred suddenly. So far, we assume that the phenomenon is not responsible for the phase transformation of the whole solder, but possibly for the local liquefaction by the exciting laser.

4. Conclusions

Using laser ultrasonics, we measured the SAW on the lead-free solder surface close to the solidus temperature. A clear change in the waveforms and the sound velocity are found between 202°C and 203°C, and also found between 208°C to 209°C. Possible explanations for the sudden change are that phase transformations wholly or locally occur in the lead-free solder.

References

1. Pavel V. Zinin, Vitali B. Prakapenka, Katherine Burgess et al.: Rev. Sci. Instrum. **87** (2016) 123908.
2 Y. Nomura, S. Minamoto and S. Nomoto: ISIJ International, Vol. 50 (2010) 1920-1924

h.sato@aist.go.jp