Monitoring of the thermal melting of a silver surface layer with use of probing laser light

V M Batenin, V V Kachalov, A V Kourilovich and V Y Mendeleyev

Joint Institute for High Temperatures of the Russian Academy of Sciences, Izhorskaya 13 Bldg 2, Moscow 125412, Russia

E-mail: v_mendeleyev@ihed.ras.ru

Abstract. Optical monitoring of the thermal melting of materials caused by high power laser radiation uses the dependence of the reflectivity on the surface temperature. The thermal melting of the surface layer is considered as the nucleation followed by the nuclei coalescence. It cannot be detected using the reflectivity. In this article, the melting of the surface layer is studied experimentally for silver samples by measuring temperature of the samples and registering patterns of probing laser light scattered and specularly reflected from the surfaces. The samples having unidirectional roughness of a surface were heated in an electric furnace. The surfaces were oriented vertically and illuminated by the probing laser beam at the wavelength of 660 nm. The power and the divergence of the incident beam were approximately 50 mW and 0.3 mrad. The study has helped clarify the features of the patterns which allow detect the melting of the surface layer. Also we found a phenomenon similar to the directional crystallization. A rod of silver grew out the molten silver against the direction of the laser beam.

1. Introduction

Laser material processing is usually accompanied by the thermal melting starting from the surface layer and propagating into the bulk of material [1,2]. Techniques to optically monitor the melting [3–5] utilize the dependence of the reflectivity on surface temperature. The reflectivity of a heated surface layer changes gradually, stops changing after the completion of the surface layer melting, and does not change during propagation of the liquid phase front into the bulk material [6–8]. Such behavior of the reflectivity does not allow to detect the nucleation and the nuclei coalescence processes during the melting of the surface layer. Therefore, the moment of time when the transition of the surface layer from its solid phase to the liquid one is complete can not be determined.

In [9], melting of silver and copper samples caused their surfaces to become unstable which, in turn, leaded to light scattering. A reason for such instability and scattering can be the nucleation and the nuclei coalescence both changing the surface texture. In this case, one can assume [10] that the melting of the surface layer can be monitored via patterns of the scattered light.

In this article we experimentally study the possibility of determining the moment of time when the transition of the surface layer from its solid phase to the liquid one is complete. An experimental setup allowed to measure the sample's temperature and to record the patterns during meltdown as it started off at the surface and followed into the bulk of material.

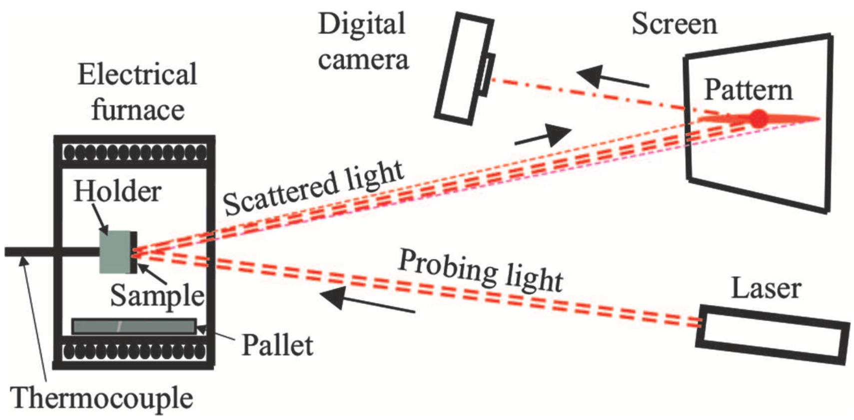

Figure 1. The schematic diagram of the experimental setup.

Sample's surface had been roughened in one direction. Such type of surface was used as an indicator of changes in roughness during the melting. The sample was heated in an electric furnace. The temperature was taken with a thermocouple and the patterns were registered with a digital camera. At the room temperature, the patterns had vivid spot of the specularly reflected beam and weak narrow stripe of scattered light. We analyzed how both the scattering and the specular reflection evolve, and defined the signs of the scattering and the specular reflection to recognize the surface layer melting processes.

2. Experimental setup and the samples

The figure 1 shows a simplified schematic diagram of experimental setup. The setup comprises a cylindrical electric furnace, a Pt-Pt/Rh thermocouple with a signal conditioner and an analog-to-digital converter, a probing laser, a screen to visualize and register the scattering and specular reflection, and a digital camera with a set of optical filters. The sample was placed in a cylindrical nickel holder. We used a semiconductor laser emitting the 660 nm wavelength light with the divergence of 0.3 mrad. The power of the laser light and the angle of incidence were approximately 50 mW and 5 degrees. The sample was heated up to $t = 970$ °C. Its temperature was recorded every second along with light patterns.

In the experiments two samples of 99.9% pure silver were studied. The samples were placed vertically in the holder having diameter of 12 mm. Sample 1 had the surface dimensions of 8 mm by 6 mm and the 0.5 mm thickness. Sample 2 had the dimensions of 9 mm by 7 mm with the same thickness. Before running the experiments, the samples had been flat ground in one direction. The ground surfaces resembled a set of mainly unidirectional grooves. Root mean square roughness measured across the grooves was 70 nm and 67 nm for samples 1 and 2, respectively. At the room temperature, the pattern exhibited vivid round spot of the specularly reflected beam and weak narrow stripe of light scattered in the direction normal to the grooves.

3. Experimental results

The dependence of the temperature on time is presented in the figure 2 for sample 1. The measured data were collected for 47 seconds and are shown at the temperatures around the melting point of $t = 960.8$ °C [11]. The root mean square error of the measured temperatures did not exceed 0.3 °C. The plot indicates that the average melting temperature was measured at $t = 961.2$ °C. As we can see from the figure, the melting of the sample started at $T = 26$ s and finished at $T = 33$ s. Note that the plot does not allow detect the transition of the surface layer from its solid phase to the liquid one.

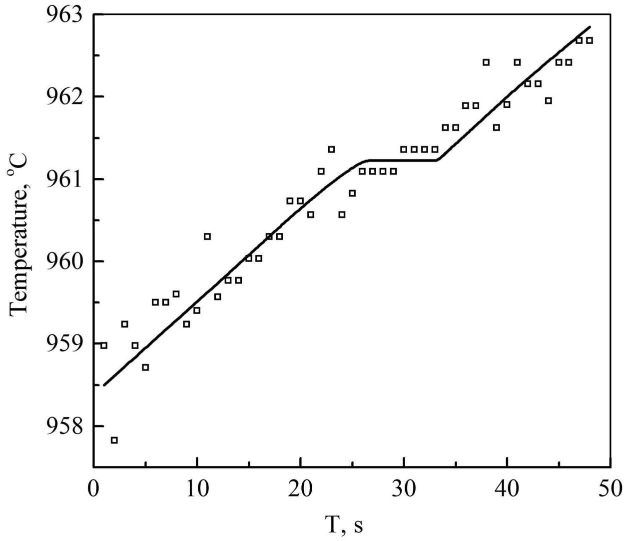

Figure 2. The dependence of the temperature on the time for sample 1. Symbol □ denotes the measured data points.

The figure 3 shows the patterns in the time range from 1 s to 41 s. The pattern taken at $T = 1$ s is quite similar to patterns found in the range from room temperature to $t = 960.5$ °C. This indicates that the roughness structure was not changing in the mentioned temperature range. As it follows from the figure, there are four distinctive behaviors of the patterns in the ranges of 24–27 s, 28–29 s, 30–32 s, and 33–37 s.

In the range of 24–27 s, the patterns taken at the 24 s, 25 s, 26 s, and 27 s moments show that the area of the unidirectional scattering becomes smaller, the isotropic scattering appears, the shape of the specularly reflected beam stays round, and the beam diameter increases. The behavior of the unidirectional scattering can be explained by smoothing out the grooves due to their melting. The divergence of the isotropic scattering is small and does not exceed 1.5 mrad. This shows that the surface is not the homogeneous roughness [10]. Such scattering could be attributed to the appearance of the surface irregularities, which are significantly larger than the wavelength of the laser light. The light scattering from such irregularities has been studied theoretically in [12] and observed experimentally in [13, 14]. Taking into account the temperature data presented in the figure 2, we can assume that the nucleation is the reason for these irregularities to appear. Increase in the specularly reflected beam diameter can be attributed to the growing curvature of the mean surface of the inhomogeneous liquid phase.

In the range of 28–29 s, the unidirectional scattering disappears, and the reflected beam shows its shape distorting and its divergence increasing. The pattern taken at $T = 28$ s shows the largest aberration. At $T = 29$ s, the reflected beam's shape aberrations become smaller. Absence of the unidirectional scattering suggests the disappearance of the roughness grooves [10]. A reason for the reflected beam's shape aberrations could be thermomechanical strains due to fixing the sample in the holder. They cause spatial irregularity of the liquid phase on the surface. This irregularity is maximum at $T = 28$ s. It reduces substantially to $T = 29$ s due to the coalescence. The increase in the divergence of the specularly reflected beam can be attributed to the curvature of the liquid phase surface [15].

In the range of 30–32 s, the specularly reflected beam's shape does not change. That is illustrated by the patterns taken at $T = 30$ s and $T = 32$ s. It can be explained by the fact that the surface layer melting is completed at $T = 30$ s and the propagation of the liquid phase front into the material starts [6].

Journal of Physics: Conference Series **774** (2016) 012122 doi:10.1088/1742-6596/774/1/012122

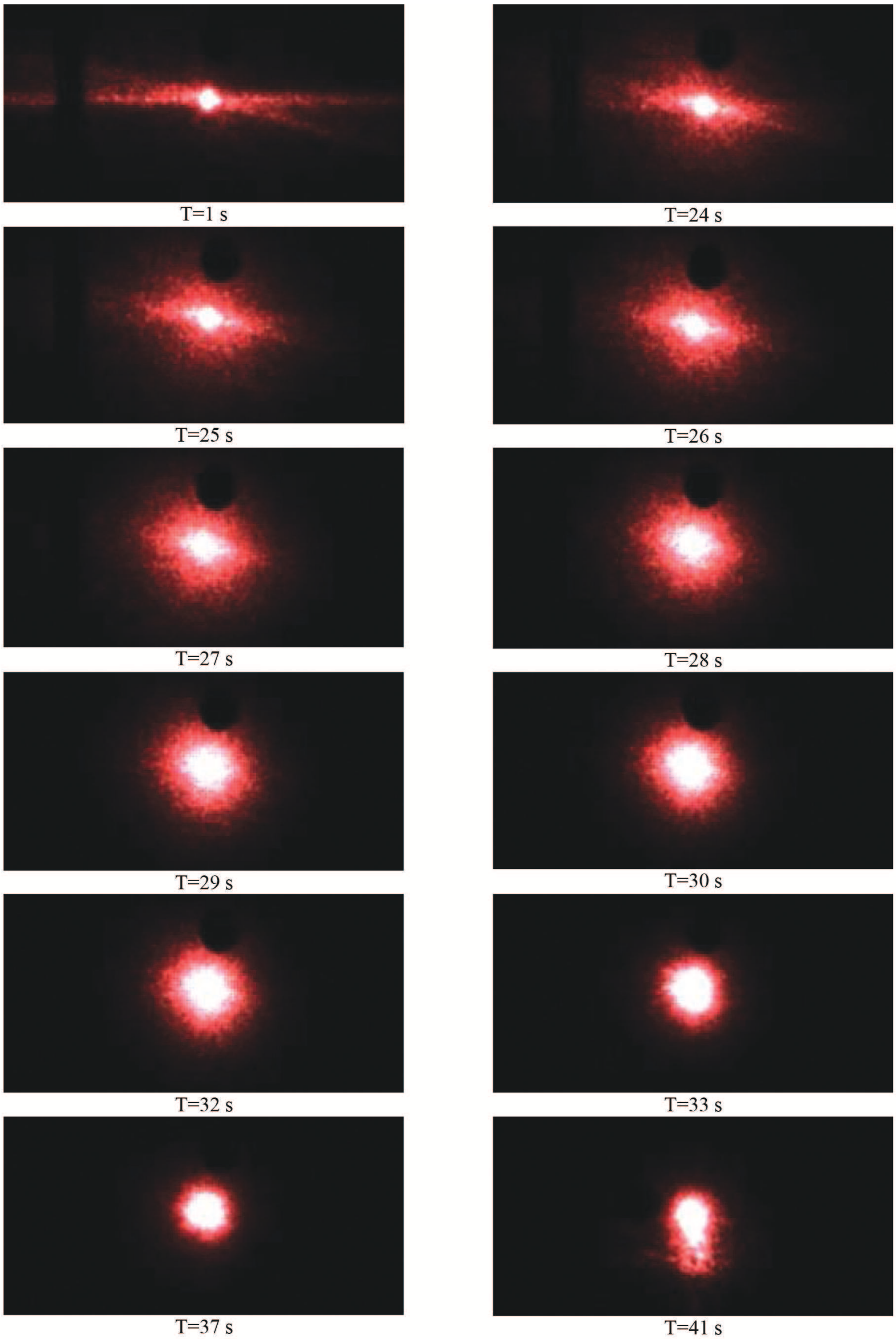

Figure 3. The patterns in the time range from 1 s to 41 s.

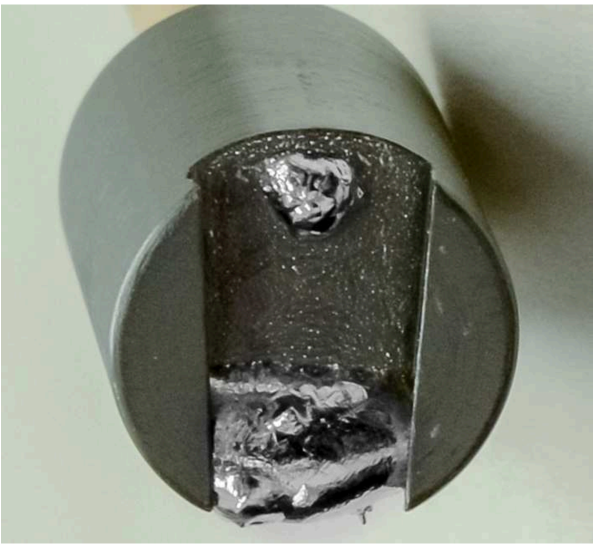

Figure 4. Frozen molten silver at sample 1.

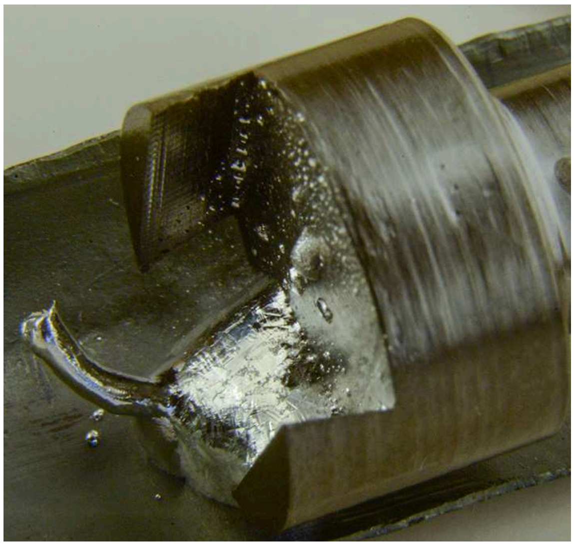

Figure 5. Frozen molten silver at sample 2.

The patterns taken at $T = 33$ s and $T = 37$ s show that the reflected beam's divergence reduces and its shape becomes round. Such transformations can be explained by molten silver spreading over the nickel substrate. The surface of the liquid phase becomes flatter, thus the reflected beam's divergence reduces. The fact that the molten silver has spread over the substrate allows us to assume the melting process completion at $T = 32$ s. Note that the time period between $T = 24$ s and $T = 32$ s corresponds to the thermal melting of the sample as defined by the use of the patterns. It agrees satisfactorily with the time period between $T = 26$ s and $T = 33$ s as defined by the use of the thermocouples.

At $T = 41$ s, the reflected beam's shape resembles a stretched drop. This is due to the molten silver leaking off the substrate under the force of gravity. The figure 4 shows frozen molten silver on the nickel substrate.

The performed study helped to clarify the following patterns' features suitable to optically monitor the thermal melting of the surface layer:

- presence of the isotropic scattering during the nucleation;
- decline in the intensity of the unidirectional scattering as a sign of the roughness evolution;
- aberrations in shape of the reflected beam as a sign of spatial irregularities in the liquid phase on the surface;
- the moment, after which the shape of the reflected beam remains unchanged, as a sign of the completion of the surface layer melting.

Unlike the measurements of the temperature and the reflectivity, the patterns of the probing light scattered and specularly reflected from a surface allow to monitor the transition of the surface layer from its solid phase to the liquid one and to determine the moment of time when the transition is complete. After this moment, the thermal melting can be studied with the use of the optical properties of the liquid phase.

A phenomenon similar to directional crystallization [16] was found for sample 2. Molten silver was leaking off the substrate onto the pallet, and a rod of the molten silver grew out against the direction of laser light. The figure 5 shows the frozen rod. Note that the rod started to grow below the incident laser beam.

4. Conclusion

We have studied experimentally the thermal melting of the silver surface layer with the use of the probing laser light. Two silver samples roughened in one direction were studied. The rough surface of each sample was oriented vertically and illuminated by the probing laser light during heating in an electric furnace. Thermocouples measured the sample's temperature and a digital camera registered the patterns of the probing light scattered and specularly reflected from the surface. At the room temperature the patterns had a vivid spot of the specularly reflected beam and weak narrow stripe of scattered light.

We have defined the following features of the patterns which allow us to detect the thermal melting of the surface layer: presence of the isotropic scattering during the nucleation; decline in the intensity of the unidirectional scattering as a sign of the roughness evolution; aberrations in the shape of the reflected beam as a sign of spatial irregularity in the liquid phase on the surface; and the moment, after which the shape of the reflected beam remains unchanged, as a sign of the completion of the surface layer melting. The study has shown a possibility to optically monitor the thermal melting of the surface layer using the light patterns.

Unlike the measurements of the temperature and the reflectivity, the patterns of the probing light scattered and specularly reflected from a surface allow to monitor the transition of the surface layer from its solid phase to the liquid one and to determine the moment of time when the transition is complete. After this moment, the thermal melting can be studied with the use of the optical properties of the liquid phase.

A phenomenon similar to the directional crystallization was observed. A silver rod grew out the molten silver against the direction of the incident laser beam.

Acknowledgments

The authors express their gratitude to M Malikov for his help in preparation of this article, and V Pilipenko for his input in creation of the experimental setup. This study has been conducted with the financial support of the Russian Fundamental Research Fund (grant No. 14-08-00696).

References

[1] Ready J F 1971 *Effects of High-Power Laser Radiation* (New York: Academic Press)
[2] Lawrence J, Pou J, Low D K Y and Toyserkani E 2010 *Advances in Laser Materials Processing: Technology, Research and Applications* (Oxford: Woodhead Publishing Limited)
[3] Bonch-Bruevich A, Imas Ya A, Romanov G S, Libenson M N and Mal'tsev L N 1968 *Sov. Phys. Tech. Phys.* **13** 640–643
[4] Zavecz T E, Saifi M A and Notis M 1975 *Appl. Phys. Lett.* **26** 165–168
[5] Downer M C, Fork R L and Shank C V 1985 *J. Opt. Soc. Am.* B **2** 595–598
[6] Anisimov S I, Imas Ya A, Romanov G S and Khodyko Yu V 1971 *Action of High-Power Radiation on Metals* (Springfield, VA: National Technical Information Service)
[7] Boneberg J, Bischof J and Leiderer P 2000 *Opt. Commun.* **174** 145–149
[8] Sokolowski-Tinten K, Schulz H, Bialkowski J and von der Linde D 1991 *Appl. Phys. A.* **53** 227–234
[9] Stabbs C M 1913 *Proc. Roy. Soc. A.* **88** 195–205
[10] Bass F G and Fuks I M 1979 *Wave Scattering from Statistically Rough Surfaces* (New York: Pergamon Press)
[11] Kikoin I K 1975 *Tables of Physical Values* (Moscow: Atomizdat)
[12] Shifrin K S 1983 *Introduction to Ocean Optics* (Leningrad: Gidrometioizdat)
[13] Born M and Wolf E 1968 *Principles of Optics* (Oxford: Pergamon Press)
[14] Sutorikhin I A, Bukaty V I, Zalaeva U I and Akulova O B 2013 *Izvestiya of Altai State University* **1** 189–193
[15] Schmid M, Zehnder S, Schwaller P, Neuenschwander B, Held M, Hunziker U and Zürcher J 2012 *ALT Proceedings* **1**(78)
[16] Scheel H J and Fukuda T 2003 *Crystal Growth Technology* (New York: John Wiley & Sons Ltd)