Lasers in Manufacturing Conference 2019

Temperature-dependent reflectivity of unpolished rolled copper for near infrared lasers

Manuel Mattern[a*], Andreas Ostendorf[a]

[a]*Ruhr-University Bochum, Universitätsstr. 150, 44801 Bochum, Germany*

Abstract

Data about the temperature-dependent reflectivity of materials are essential for the modeling of laser-material interactions. Most of the calculated and experimentally acquired data currently available for copper consider only polished and oxygen free copper surfaces. However, the surface of industrially processed copper is usually unpolished, therefore having a slightly oxidized surface. In this paper, the reflectivity of unpolished rolled copper foils for 1064 nm wavelength is measured for temperatures up to 800 °C, using an integrating sphere. Measurements with recently polished copper are conducted for reference. The measurements for the unpolished copper show a behavior of the reflectivity over temperature which is completely different from the one for the polished copper samples. After cooling, the reflectivity for the unpolished samples is increased by about 2.5 percentage points compared to the initial values. This shows the necessity to measure the reflectivity also of oxidized samples when modeling a real process.

Keywords: reflectivity; copper; temperature; near infrared; oxidation

1. Introduction

The reflectivity of materials plays an important role in laser material processing, as only the absorbed fraction of the incident laser power is available for driving the desired process. Despite the development of green and blue lasers, solid-state lasers having a wavelength shortly above one micron are still the predominant beam source for laser material processing of metals. The accuracy of reflectivity data is of special importance for process models involving highly reflective metals like copper, since small errors can lead to substantial changes in the process results. Reflectivity data for copper at room temperature are

* Corresponding author. Tel.: +49-234-3223452; fax: +49-234-3214259.
E-mail address: mattern@lat.rub.de.

available for all relevant laser wavelengths. However, the temperature of the surface is usually increasing during laser material processing. For this reason, knowledge about the temperature dependence of the reflectivity is required. Ujihara, 1972 calculated the reflectivity of copper over temperature up to the melting point, based on the Drude theory and the theory of electron-phonon collision. Blom et al., 2003 show calculated data points for copper at 1064 nm wavelength, including one at a temperature of 2000 K. However, theoretical calculations may deviate from the reflectivity of real materials, especially if the surface features unconsidered imperfections like surface roughness or oxidation. The following publications report on different approaches to measure the reflectivity of samples in an integrating sphere.

Chan et al., 1977 presented a study in which the reflectivity of copper for ruby lasers was measured using an integrating sphere. The reflectivity was measured with the same laser pulses that heated the sample. Since the surface temperature of the sample was not measured but only calculated on the basis of the emitted laser energy, deviations of parts of the measurement points from the Ujihara theory were attributed to unintended local melting and flow of the copper surface.

Zhang and Modest, 1998 measured the reflectivity of ceramics up to their decomposition temperature. In their approach, the samples were heated inside an integrating sphere by the help of a CO_2 or Nd:YAG laser, while a small focused probe laser was used for the reflectivity measurement. A high-speed pyrometer recorded the surface temperature of the sample. The heat radiation emitted from the heated zone and the reflected power from the heating laser were eliminated from the measurement by the help of a bandpass filter and an optical chopper in combination with lock-in amplifiers for each of the two photodetectors.

Mann et al., 2014 conducted measurements of the absorptivity of copper and copper alloys for three different wavelengths, including 1064 nm. The laser was only used for the reflectivity measurement, while the sample was heated to temperatures up to 500 °C by a heating plate. A thermocouple was used to measure the surface temperature of the sample. Unfortunately, despite the use of argon as shielding gas, the absorptivity was irreversibly increasing due to oxidation when the temperature exceeded 350 °C.

This study presents a new experimental setup for measuring the reflectivity of copper samples, tanking into account the advantages and disadvantages of the above-mentioned approaches. The measurements are conducted on unpolished samples for a wavelength of 1064 nm and for temperatures up to 800 °C. The discussion does not only focus on the measurement results, but also takes into account the suitability of the experimental setup for the measurements.

2. Experimental setup and methods

To measure the temperature-dependent reflectivity of the samples, an integrating sphere was used. The sphere, which is depicted in Fig. 1 (a), is coated with barium sulfate ($BaSO_4$), has a diameter of 500 mm and is equipped with a heating plate in its center. By choosing this location, the impact of the heat on the optical coating of the sphere can be kept low. The stainless steel plate separating the copper from the heating plate is used to protect the heating plate in case of accidental wetting with liquid copper due to overheating. A thermocouple on the surface of the sample provides the temperature signal required by the microcontroller (not depicted) for temperature control. The sphere is also equipped with a camera for surveillance and a gas

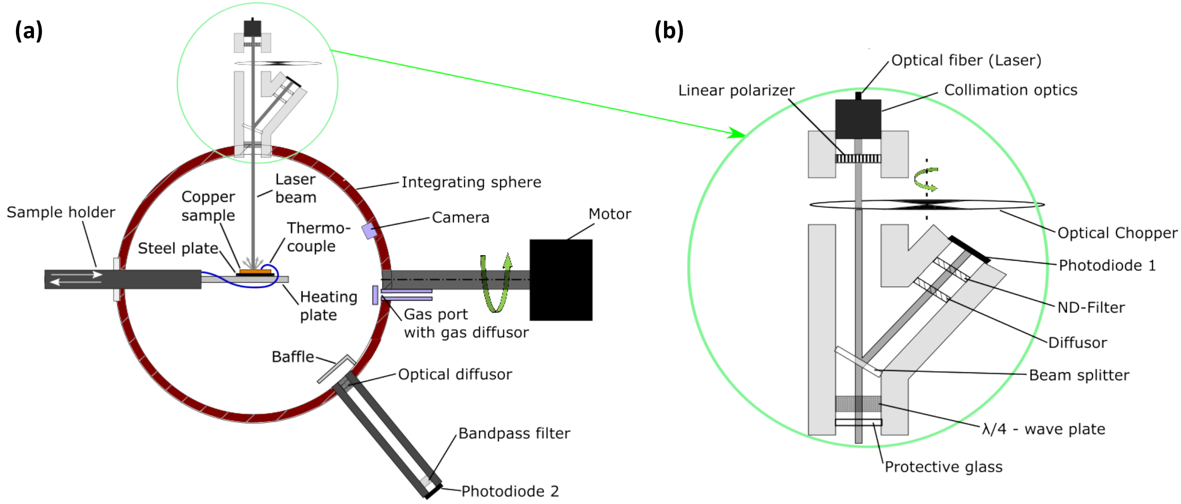


Fig. 1. (a) Setup of the integrating sphere with heating plate and (b) detail view of the optical tower

port for the delivery of shielding gas. The laser beam (1064 nm) is delivered through an optical fiber and collimated at the top of the optical tower (Fig. 1 (b)). In this optical tower, the laser beam first passes a linear polarizer and then an optical chopper. The linear polarizer is essential to stabilize the splitting ratio at the beam splitter, which is located below the optical chopper. A comparable small part of the laser radiation is reflected onto photodiode 1 (PD1), after passing a diffusor and a neutral density filter to create an appropriate illumination of the diode. The greater part of the laser radiation, however, passes the beam splitter, is then circularly polarized by a λ/4 wave plate and finally enters the sphere through an optical window. In the sphere, the laser beam hits the sample surface at an angle of 10 °. This angle minimizes the losses of reflected light back into the optical tower. It is also similar to most scenarios in laser processing of copper, where the processing optics have to be protected from back reflections.

Photodiode 2 (PD2) is used to determine the amount of light which is reflected by the sample. The opening to the photodiode is protected against direct illumination by a baffle. Furthermore, the opening is placed in the lower half of the sphere to further decrease the dependence of the signal for PD2 on the direction of the initial reflections. To reduce measurement errors caused by the thermal radiation emitted by the heating plate, a bandpass filter for 1064 nm transmission wavelength is placed in front of the photodiode. The filter is placed at the end of a black anodized tube, which absorbs light for incident angles higher than 6 ° and thus prevents a blue shift of the transmission window. However, the filter cannot block heat radiation within the transmission bandwidth. For this reason, the laser beam is periodically blocked by the optical chopper in the optical tower, which has already been mentioned above (Fig. 1 (b)). This way, the amplitude of the background signal can be measured and deducted from the amplitudes I_{PD1} and I_{PD2} of the complete signal for the photodiodes (principle of a lock-in amplifier). The resulting processed signals I_{PD1^*} and I_{PD2^*} for the photodiodes are used to calculate the reflectivity of the sample independently from fluctuations of the laser power. The uncalibrated ratio

$$R_{uncal} = \frac{I_{PD2^*}}{I_{PD1^*}} \tag{1}$$

is proportional to the reflectivity R of the sample.

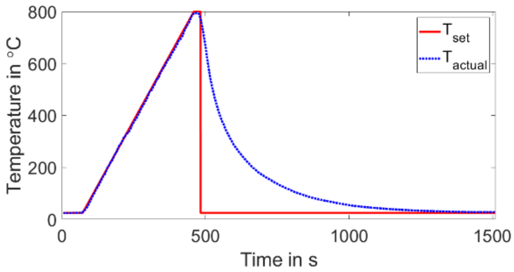


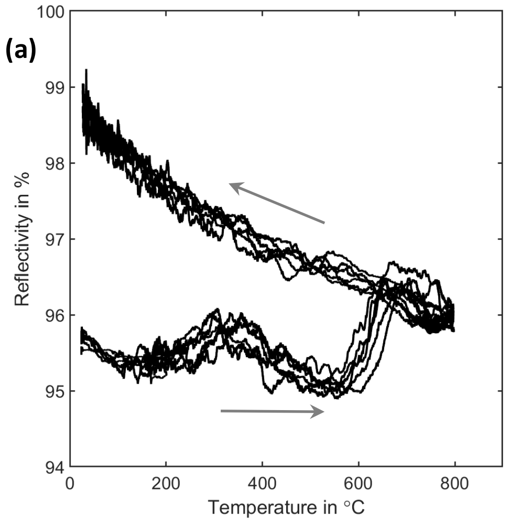
Fig. 2 Temporal behaviour of the set temperature T_{set} and the actual temperature T_{actual}

The measurements are divided into two phases. In the first phase, the initial reflectivity of the samples at room temperature is determined. To reduce possible measurement errors, the ratio R_{uncal} for the samples at room temperature is compared to the same ratio of a reference sample of known reflectivity, which was recorded just before and after each sample for calibration purposes. To reduce the influence of noise, the measurements on the sample and on the reference are averaged over one minute. In the second phase, the reflectivity is measured over temperature. For this measurement, after introducing the sample into the sphere, the sphere is flushed with the shielding gas for 10 minutes. Subsequently, the measurement is started. The red line in Fig. 2 is representing the temporal temperature profile which is set by the microcontroller. At first, the signals of the photodiodes are recorded for one minute at room temperature, which delivers sufficient data for a stable calibration. Then the sample is heated at a specified heating rate up to 800 °C. After holding the maximum temperature for 10 seconds, the heating plate is switched off and the sample cools down to room temperature. The shielding gas is kept flowing throughout the measurement in order to keep the heating of the optical coating of the sphere low.

3. Results and discussion

The initial reflectivity of the untreated copper samples in this study is between 95.53 % and 95.93 %. The shielding gas is nitrogen. Fig. 3 (a) shows the measurement results for six samples that were heated at a heating rate of 2 °C / s. For the reason of slightly different surface conditions from sample to sample, the measurements start at different initial reflectivity values; however, the main course of the curves is quite similar, demonstrating the reproducibility of the measurement. All curves start with a slight reduction of reflectivity until a temperature of about 150 °C is reached. In the following, until a temperature of about 350 °C, the reflectivity rises again by about 0.5 percentage points. From about 350 °C up to about 550 °C, the reflectivity decreases by nearly 1 percentage point, followed by an increase of more than 1 percentage point until a temperature of about 650 °C is reached. When heating the sample further up to 800 °C, a slight decrease of the reflectivity is visible in the plot. At the maximum temperature of 800 °C, the reflectivity is a little higher than at room temperature before heating. When cooling down, the reflectivity rises by about 2.5 percentage points to a value of about 98.5 %.

At least for the heating phase, the recorded measurements in Fig. 3 (a) are different than expected from literature, where the reflectivity for pure copper decreases with increasing temperature. However, for the cooling phase the trend of the reflectivity is as expected. Reflectivity data from Blom et al., 2003 are shown in Fig. 3 (b) for comparison. The measurements of this study were not conducted for pure copper samples, but for slightly oxidized copper surfaces, and show a good reproducibility. The total increase of the



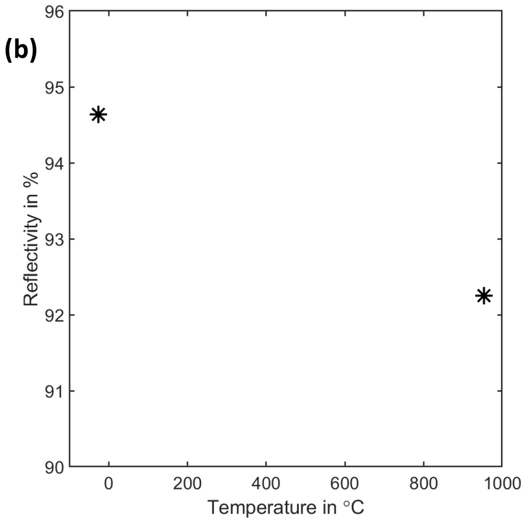


Fig. 3. (a) Measured reflectivity over temperature for untreated copper samples and (b) Reflectivity over temperature for copper according to Blom et al., 2003

reflectivity by more than 2.5 percentage points from the beginning to the end of the measurement indicates an irreversible modification of the sample during the heating and cooling cycle.

For the results in Fig. 4 (a), three consecutive heating and cooling cycles were conducted for three samples each. The reflectivity over temperature has a completely different characteristic for the second and third cycle than for the first one. While the first cycle reveals an irreversible process, the second and third cycle show a completely reversible behavior, which corresponds to the characteristics known from literature. However, in industrial laser materials processing the material is usually used as delivered. For this reason, the behavior of the reflectivity during the first heating process is the most interesting one for the modeling of laser material processing.

Fig. 4 (b) shows again three heating and cooling cycles on the same sample. However, this time the sample was polished down to a 1 µm diamond suspension before the first heating process. The measurement now starts at a reflectivity of 97.3 %. During the first heating phase, irreversible changes only take place up to a temperature of about 350 °C. From then on, the reflectivity decreases by about 2 percentage points until the maximum temperature of 800 °C is reached. When cooling down, the reflectivity increases along the same straight line as it had decreased before until room temperature is reached. The total reflectivity after cooling is about 98.5 % and thus increased by about 1 percentage point compared to the initial value before the first heating. The second and third heating and cooling cycles are comparable to the ones of the unpolished samples.

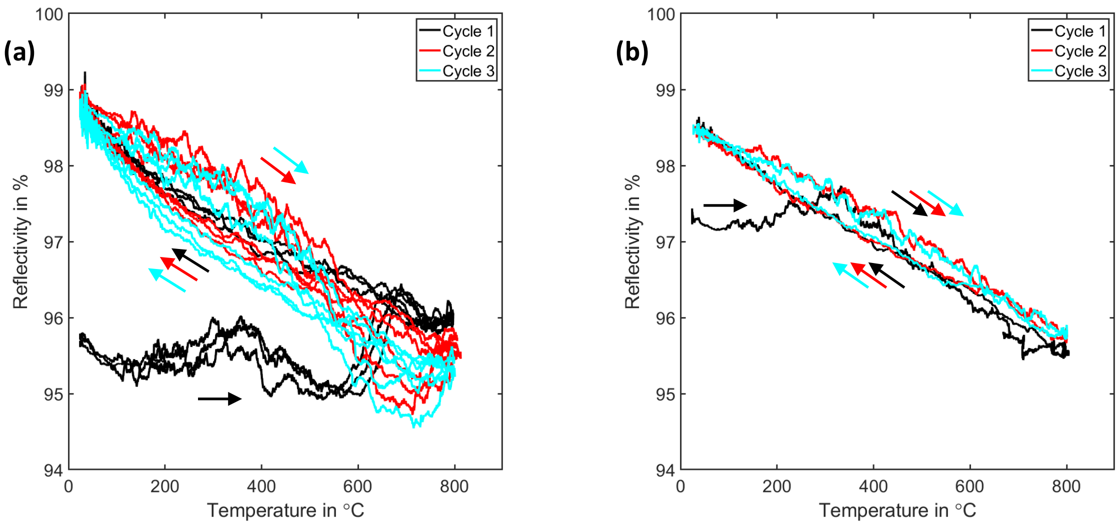


Fig. 4. Reflectivity over temperature for three consecutive heating cycles 1-3 (a) on each of three untreated samples and (b) on one polished sample

4. Conclusion

The results show that the reflectivity of real samples over temperature can deviate dramatically from the theoretically calculated values. The trend of rising or falling reflectivity might in parts even be inverse if compared to the theory.

By the help of the integrating sphere used in this study, the reflectivity of the samples could be measured down to an estimated precision better than 0.1 % at room temperature. The change of reflectivity during the heating and cooling cycle could also be tracked for all samples. However, heating of the samples also lead to indirect heating of the sphere. Although the temperature of the sphere does not change dramatically, the integrating behavior is influenced noticeable due to the multiple reflections on the coating of the sphere. This is why the accuracy of the reflectivity-measurement is reduced during dynamic measurements for temperatures up to 800 °C. Nonetheless, despite the problem that the temperature of the sphere affects the measurement, certain characteristics could undoubtedly be proven. One of these characteristics is an increase of the reflectivity by about 2.5 percentage points for the untreated samples and by about 1 percentage point for the polished sample between the initial reflectivity and the reflectivity after heating. It could also be demonstrated that the initial surface condition has only little effect on the final reflectivity of about 98.5 % after cooling down from 800 °C. This result is higher than expected from Blom et al., 2003 , but they show good agreement with the measured absorptivity of 1.5 % at room temperature in Duley, 1986. Since the reflectivity of the integrating sphere slightly decreases for increasing temperatures, the measured increase of reflectivity during the first heating can only be caused by the sample.

This study shows that the measurement of the reflectivity over temperature can be essential for a realistic model of industrial laser processes. Especially for the demonstrated example of unpolished rolled copper, a comparison of the reflectivity before and after the temperature treatment reveals a reduction of the absorptivity by about 65 %. To allow a high precision also for measurements at elevated temperatures, the authors suggest to equip the sphere with a temperature management system in order to keep the integrating behavior of the sphere constant.

Acknowledgments

We want to thank the German Research Foundation (DFG) for funding the project under Grant OS-188/37-1.

References

Blom, A., Dunias, P., van Engen, P., Hoving, W., Kramer, J. de, 2003. Process spread reduction of laser microspot welding of thin copper parts using real-time control, in: High-Power Lasers and Applications, San Jose, CA. Saturday 25 January 2003. SPIE, pp. 493-507.

Chan, P.W., Chan, Y.W., NG, H.S., 1977. Reflectivity of Metals at High Temperatures Heated by Pulsed Laser. Physics Letters 61 (3), 151-153.

Duley, W.W., 1986. Laser Material Interactions of Relevance To Metal Surface Treatment, in: Draper, C.W., Mazzoldi, P. (Eds.), Laser Surface Treatment of Metals, vol. 6. Springer Netherlands, Dordrecht, pp. 3-16.

Mann, V., Hugger, F., Roth, S., Schmidt, M., 2014. Influence of Temperature and Wavelength on Optical Behavior of Copper Alloys. AMM 655, 89-94.

Ujihara, K., 1972. Reflectivity of Metals at High Temperatures. J. Appl. Phys. 43 (5), 2376.

Zhang, Z., Modest, M.F., 1998. Temperature-Dependent Absorptances of Ceramics for Nd: YAG and CO2 Laser Processing Applications. J. Heat Transfer 120 (2), 322-327.