Plasma Induced On Indenter Balls

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Abstract

There is an increasing demand to enable high throughput experimentation to characterize and develop new materials in a very short time. The investigated hardness measurement method differs from conventional hardness measurements in how the force is applied. The new method is based on laser-induced shockwaves. A shockwave is created with a nanosecond pulsed TEA CO_2 laser on top of an indenter. The pressure of the shockwave is used to push an indenter inside a workpiece. A quadratic laser focus area of 4 mm², having a diagonal larger than the indenter diameter, leads to interactions of the laser beam with the surrounding material, which affects the plasma formation and results in heating of the material underneath. Material heating decreases the yield point and accordingly the hardness. Therefore, influence of pulse energy and plasma formation on heating of material are investigated to understand the interaction between the high intensity laser beam, the indenter and the material underneath. It is shown that a 3 mm indenter diameter reduces the maximum estimated temperature of the workpiece (X5CrNi18-10) underneath down to 64°C. With an additional positioning unit in combination with indenter diameter of 3 mm or larger no significant heat input was obtained anymore in the workpiece underneath.

Keywords

Hardness, Laser, Condition monitoring

1 Introduction

Conventional material developments are based on costly experimental investigations of different material properties. To meet the demands of efficient identification of compositions, which pursue the objective to realizing a specific performance profile of a material, high-throughput experimentation in materials science has been recognized as a new scientific approach to generate new knowledge (Potyrailo et al., 2011). For also achieving high through-put material characterization, new measurement methods must be found. New techniques must operate at least at the same accuracy level as existing conventional methods. A conventional material characterization method is the hardness measurement. However, the indentation and evaluation process during these hardness measurements usually take several seconds. In this paper, a new hardness measurement method is presented and analyzed, which is based on laser induced shockwaves. If energy density of CO_2 laser pulse exceeds a critical point, a plasma is induced and will result in a shockwave (O'Keefe et al., 1973). According to Walter et al. (2007), the shockwave expands spherically. The expanding shockwave pushes an indenter inside a test material. The depth and diameter of the indentation are evaluated. In the context of high through-put, laser systems and laser based processes offer high flexibility and high dynamic. Laser systems are used as measuring devices since the 1960s. Brech and Cross (1962) first introduced material emission analysis with a ruby laser. Amongst others, pulsed CO_2 lasers are used for the analysis and processing of samples because pre-treatment (Veenaas et al., 2013) and ablation layers are not necessary (Miziolek et al., 2006). The unwanted effect of material removal during the creation of the shockwave can be avoided if higher wavelengths are applied. Vollertsen et al. (2009) showed for Aluminium that no ablation was observed during the processing with TEA CO_2 laser. This is because almost all the laser irradiation is absorbed by plasma (Bergmann and Hügel, 1998). Additionally, standard atmosphere conditions are indispensable for high through-put industrial measurement applications. Marpaung et al. (2000) analyzed the influence of atmosphere on CO_2 laser induced plasma. They still detected in standard atmosphere conditions the characteristic plasma regions. One part is the primary plasma, which is found above the surface. The primary plasma is the initial explosion energy source for the shockwave (Marpaung et al., 2000). The other part is the secondary plasma, which expands away from the primary plasma. Vollertsen et al. (2009) observed that for forming operations of thin metal sheets shockwave pressure in the range of some megapascal are necessary. Experiments showed that the pressure is decreasing regressively with increasing distance (Czotscher and Vollertsen, 2017) and that the plasma geometry correlates with the pressure (Czotscher et al., 2017). Veenaas et al. (2013) showed that for micro forming operations CO_2-lasers are a reliable tool. The accuracy of the positioning system between standard specimens, laser beam and workpiece is of high importance but also the conditions that affect the process. Radziemski et al. (1985) conducted experiments with pulsed CO_2 laser in ambient air and measured the plasma temperature. They showed that plasma temperatures of up to 19000 K are observed after 1 µs. The temperature reduces to 11000 K after 25 µs. Heating of material reduces the yield point and influences accordingly the hardness of the material. The goal of this paper is to understand thermal radiation by plasma and laser beam to adjust the set-up accordingly and limit these influences for the indentation process. The influence of the laser beam and the plasma on the workpiece temperature is analyzed. Additionally, it is estimated how the laser beam and the plasma affect the temperature in the indenter.

2 Experimental set-up and methods

The experiments were conducted with a pulsed TEA CO_2 laser. The laser has pulse duration of 100 ns and a maximum energy per pulse of 6 J. For the experiments, up to 6 J pulse energy was used. The schematic experimental setup is shown in **Fig. 1**. The pulsed laser beam was irradiated on ceramic (Al_2O_3) indenters with a diameter between 1 mm and up to 5 mm. Indentations were created on rolled and annealed steel (X5CrNi18-10). The indentation depth and diameter was measured with the laser-scanning microscope VK-9710 from Keyence. The 50-fold objective lens has an accuracy of 14 nm at the height difference of 2 µm. The measurement accuracy is 1.9 µm at a 1000-fold magnification.

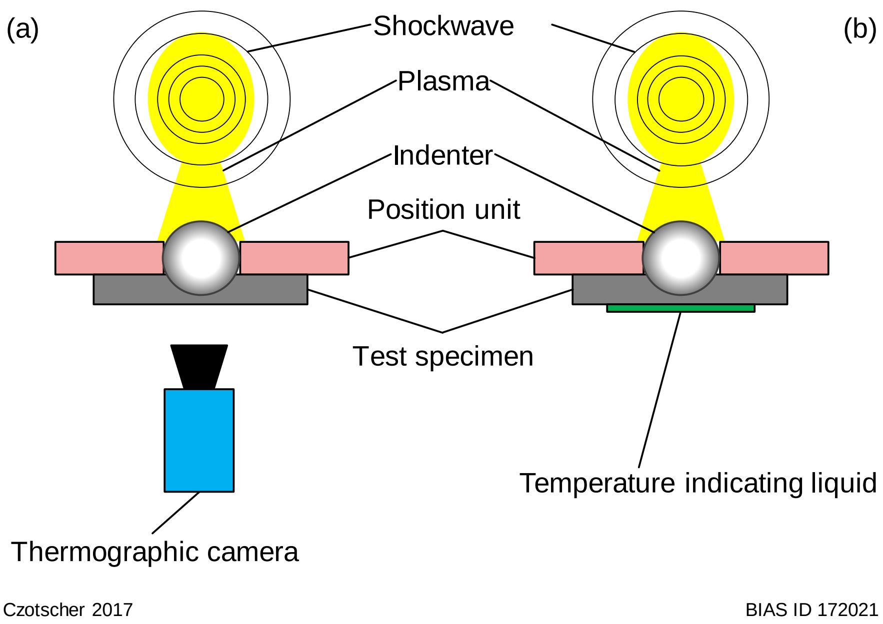

Figure 1: *Schematic experimental setups: (a) with thermographic camera and (b) with indicating liquid*

For capturing the plasma, a neutral absorber was placed between the camera Prosilica GT 2450 from Allied Vision and plasma. This was done to reduce the intensity of the plasma emission. The formation and the maximum expansion of the primary plasma was captured with the camera. The camera has a resolution of 2447 * 2050 pixels and a maximum framerate at full resolution of 15 fps. The exposure time can be varied between 25 µs and 42.9 s. For the experiments, the lowest exposure time was chosen. The camera was triggered with the TEA CO_2 laser.

Temperatures are measured with the thermographic camera VarioCAM hr head from InfraTec on the backside of rolled steel sheets (X5CrNi18-10), as shown in **Fig. 1**. The recording frequency of the camera is 60 Hz. The measured temperatures were compared to temperature indicating liquids Omegalaq, from Omega, which dry at room temperature and melt at a defined temperature, which changes their colour. The response time of the lacquers is 1 ms. Air-drying lacquers were used, which melt at 97°C, 107°C, 135°C, 149°C, 177°C, 191°C and 204°C.

When heating of the material is only caused by laser irradiation and an adiabatic process is assumed, the heat input of the laser beam ΔQ_w in the material can be calculated as follows:

$$\Delta Q_w = c_p \cdot m_W \cdot \Delta T \qquad (1)$$

The specific heat capacity is c_p, ΔT is the increase in temperature and m_W the heated mass. The mass heated by the laser is calculated as follows:

$$m_w = A_f \cdot t_p \cdot \rho \qquad (2)$$

A_f is the focus area of 4 mm² of the laser beam, ρ is the material density and t_p is the thermal penetration depth of the laser beam, which can be defined as:

$$t_p = \lambda / (4 \cdot \pi \cdot k) \qquad (3)$$

The **Eq. 3** is valid for short pulse durations and if melting is not present. By adding **Eq. 2**, **Eq. 3** in **Eq. 1** the heat input in the material can be calculated as:

$$\Delta Q_w = c_p \cdot A_f \cdot \lambda / (4 \cdot \pi \cdot k) \cdot \rho \cdot \Delta T \qquad (4)$$

The thermal penetration depth determines the depth until the laser intensity is reduced significantly in the material and causes no further significant interaction. The wavelength of the laser is $\lambda = 10.6\ \mu m$ and k is the extinction coefficient, which is a dimensionless quantity describing the attenuation of a certain wavelength within a material. Properties found in the literature of the analysed materials are shown in **Table 1**.

Material	**k**	**ρ**	c_p
Al99.5	86.0	2.70 g/cm³	0.90 J/(g·K)
Alloyed steel	20.5*	7.90 g/cm³	0.46 J/(g·K)
Al_2O_3	0.2	3.94 g/cm³	0.90 J/(g·K)

Table 1: *Properties of the tested materials (*values derived from iron)*

3 Results

Fig. 2 shows the formation of plasma. The plasma grows to a large extent the first 4 µs. Afterwards the expansion rate decreases until 6 µs. For further analysis, the expansion at formation time of 6 µs was selected for the experiments.

Fig.3 shows the maximum extension of the plasma created on different indenter diameters. On 3 mm indenter diameter, the plasma is fully induced by the laser beam only on top of the active tool. Instead, for 1 mm and 2 mm indenter diameters, the laser beam irradiates also at the side of the indenter and on the workpiece underneath. Accordingly, a diagonal of the laser spot larger than the indenter diameter leads to interactions of the laser beam with the surrounding material.

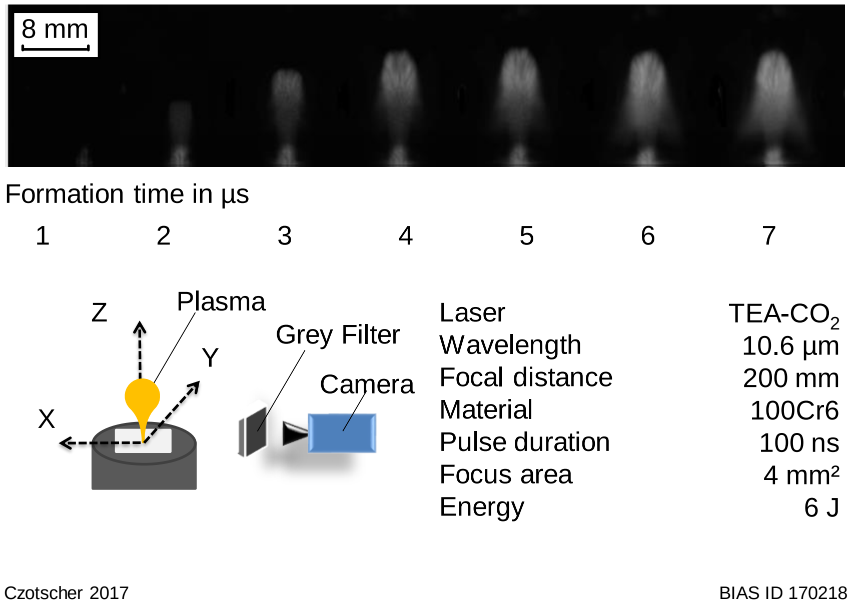

Figure 2: *Formation time of plasma on steel*

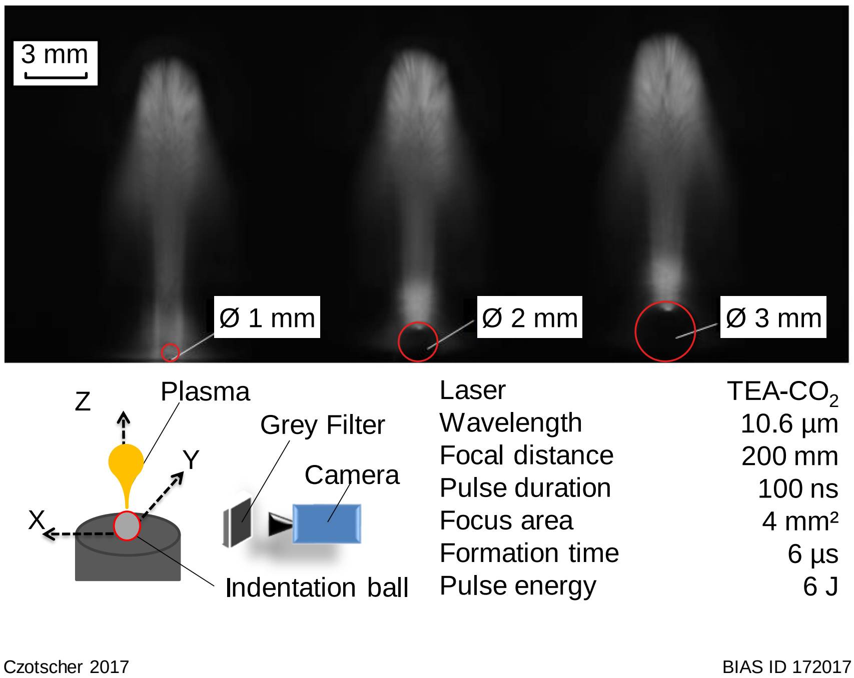

Figure 3: *Formation of plasma on top of indenter*

The temperatures, which develop in the metal sheets, are shown in **Fig. 4** for different set-ups. The temperatures are measured on the backside of the sheet and increase exponentially for lower sheet thicknesses. The results of the thermal camera and temperature indicating lacquer are in good accordance with the thicker sheets of 25 µm and 50 µm. Instead, on the backside of the 10 µm sheet, the lacquer still melts at 191°C, but not at 204°C anymore. In this case, only 154°C was measured with the thermographic camera. A larger indenter diameter leads to a decrease in temperature. For the 1 mm indenter diameter in combination with a positioning unit a significant increase in temperature is observed on the 25 µm sheet. In the thinner sheets, up to 50 µm, no temperature increase is observed with the 3 mm indenter diameter in combination with the positioning unit. However, a small temperature increase is observed in the 100 µm sheets for the 1 mm and 3 mm indenter with positioning unit.

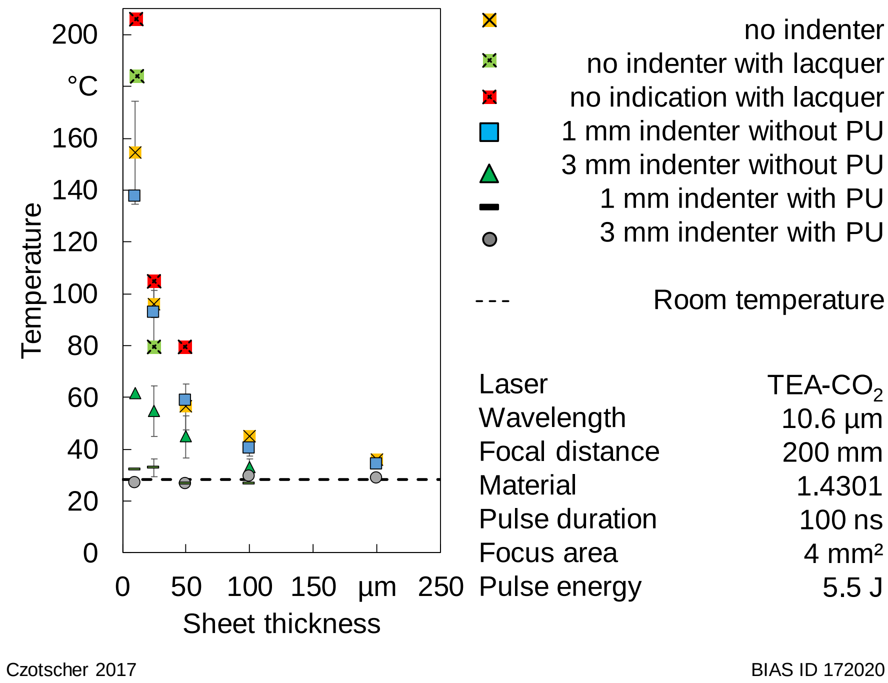

Figure 4: *Measured temperatures on steel sheets with different indenter diameters and positioning units (PU)*

Additionally, different sheets are placed laterally to the laser impact area. The temperatures were again measured on the backside of different sheet thicknesses to identify the influence of plasma on heating. The temperature field induced by the plasma, shown in **Fig. 5**, can be compared with the plasma plume shown in **Fig. 3**. Temperatures up to 50°C are measured on the backside of 10 µm steel sheets **Fig. 6**. The lacquer, which melts at 79°C, did not show any effects on 10 µm sheets. Thus, the lacquer was faced towards the laser impact area, on the front side of the sheet. In this case, direct interaction of the expanding plasma and the lacquer did also not show any effects.

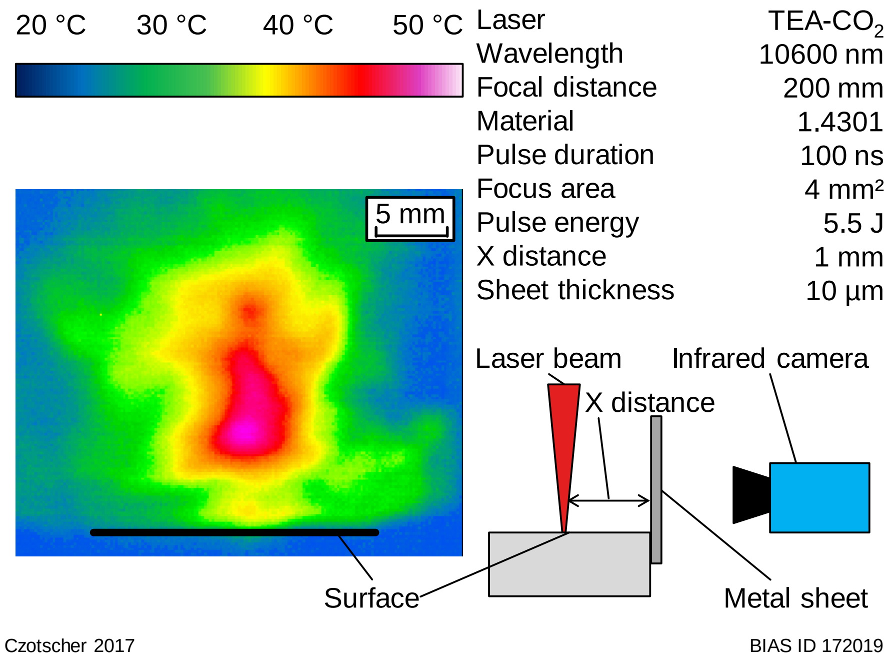

Figure 5: *Temperature field measured on the back side on 10 µm thick metal sheet*

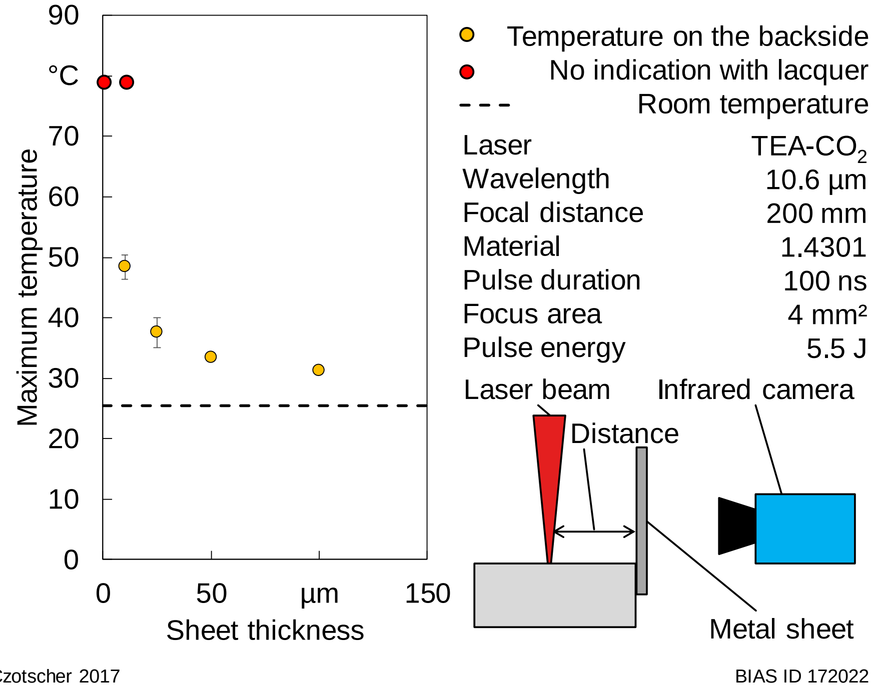

Figure 6: *Measured temperatures on the back side of different steel sheet thicknesses*

4 Discussion

The conducted experiments showed that the laser focus area, whose diagonal is larger than the indenter diameter, influences the temperature of the material underneath the indenter. Expansion of the high temperature plasma leads to additional heating of the material, as it is shown in **Fig. 5**. Due to the high intensity of the laser beam and expanding plasma, heating is relevant for laser-induced shockwave processes. However, depending on the indenter diameter, which is placed between the radiation of the laser beam and the test material, heating of the test material is significantly reduced.

The interaction of the high intensity laser beam with different workpieces did not lead to an ablation of the material on steel (X5CrNi18-10). This was already shown by Miziolek et al. (2006). The reason for this is the effect of inverse Bremsstrahlung, which leads to an absorption of laser energy of the emitted electrons. The absorption increases proportionally with the square of the wavelength. Therefore, almost all the laser irradiation is absorbed by the plasma (Bergmann and Hügel, 1998). However, a temperature increase of 152°C on top of the steel sheet caused by only laser irradiation, is a result of energy consumption of 0.091 mJ by the steel material (for calculation see **Table 1**, **Eq. 4**). The energy consumption of the material divided by a laser pulse energy of 5.5 J, shows that in this case 0.002% of the laser beam is still absorbed and transferred to heat inside the material. Thus, heating during the forming process cannot be excluded for indenter diameters smaller than the diagonal of the laser spot. The temperature can be reduced with an additional positioning unit. For high throughput characterization, heat accumulation may still impact the material properties for smaller diameters, as it is demonstrated in **Fig. 4**. Moreover, the formed plasma at the side of a 2 mm or 1 mm indenter could cause a shockwave, which counteracts the movement (as it is shown in **Fig. 3**). This can also influence the reproducibility of the indentation process.

Instead, a larger indenter diameter reduces the temperature of the workpiece underneath. When the indenter diameter is larger than the laser spot diagonal, only heating by the plasma is relevant. Heating of the whole indenter due to the laser for indenter diameters larger than 2 mm can be neglected. Because no melting was observed on the indenters, a temperature increase of less than 2072°C can be assumed. The laser beam irradiation within the pulse duration of 100 ns causes therefore a maximum heat input by the laser beam of 123 mJ (for calculation see **Table 1**, **Eq. 4**). This heat input would lead to a total temperature increase of 8.6 K in the whole 2 mm indenter.

For an indenter diameter of 3 mm, the temperature on the workpiece underneath is decreased about 50% down to 64°C. If no positioning unit is placed the plasma can expand along the indenter and leads to a heating of the material underneath (as shown in **Fig. 5**). With an additional positioning unit in combination with an indenter diameter of 3 mm or larger no significant heat input was obtained anymore in the workpiece underneath the indenter.

5 Conclusion

The present study explained that an indenter diameter larger than the diagonal of the laser spot area reduces the maximum estimated temperature of the workpiece (X5CrNi18-10) underneath. With an additional positioning unit in combination with an indenter diameter of 3 mm or larger no significant temperature increase was obtained anymore on the workpiece underneath the indenter. The additional positioning unit shields from heating caused by plasma, whereas indenters with a larger diameter shield the workpiece underneath from heating caused by laser irradiation.

5.1 Acknowledgments

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