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LASER SURFACE MELTING OF COBALT MONOCRYSTAL

The paper presents a concise description of laser surface melting of cobalt monocrystal and some related phenomena. Pulses of electromagnetic radiation of nano and mili-second duration, generated by Nd:YAG laser were used for surface heating. Light was incident on (0001) plane of cobalt monocrystal. The influence of surface roughness on the domain image observed with SEM (type I magnetic contrast) was examined. Roughness of the surface was changed by polishing with diamond powders and superficial melting with nanosecond pulse of laser energy. The r.m.s. profile deviation σ of the examined surfaces was determined with reflexometric method. The depth of layer melted with nanosecond light pulse was evaluated for the isotropic model of metal and on the assumption that material constants are not temperature-dependent. Qualitative discussion of the phenomena associated with laser surface melting, i.e. generation of capillary waves and refinement of the Kittel type magnetic domains was included.

Keywords: laser, cobalt monocrystal, superficial melting, capillary waves, Kittel type magnetic domains.

1. INTRODUCTION

Laser surface melting of cobalt monocrystal is a special case of wider researches on superficial melting and related phenomena which have been conducted by Wojtczak and his group for many years [1-4]. These researches included both theoretical description and modelling [1,3] as well as experimental examinations [2,5] of superficial melting of solids with conventional methods (when the sample is slowly heated and phase changes are observed on its surface).

Examinations of superficial phase changes for the crystalline, magnetic materials are especially interesting [3,5-9], because in such materials phase changes both of the first and the second kind can take place. Laser heating, when compared with conventional method, can be very quick (duration of the pulse of suitable energy can be even shorter than one nanosecond) and can be applied locally, while the rest of the sample has the temperature of its surroundings.

The aim of this paper is to present brief and concise description of examination methods and results of the experiments with pulsed laser heating and phenomena which occur on the heated surface of cobalt monocrystal (for (0001) plane). Some of these phenomena were presented in detail in previous papers [7-9].

Cobalt monocrystal can appear in the close-packed hexagonal phase (hcp) and in the body-centred-cubic phase, characterized with different arrangements of atoms. Transition between these two structures (called martensitic transition) is observed for precisely determined temperature T_m. Transition between ferromagnetic and paramagnetic phase occurs for the Curie temperature T_c, which is higher than T_m. In ferromagnetic phase one can expect the definite structure of magnetic domains [10].

Strong magnetic anisotropy is observed along direction [0001] for hexagonal phase. It gives the origin to magnetic field of considerable strength on the outer side of the plane perpendicular to the axis of anisotropy. It means that conditions of creation of the Kittel type open magnetic domains are fulfilled. Surfaces of the samples with this kind of magnetic domains structure produce type-I magnetic contrast in SEM observations [11]. This type of contrast is reduced by topographic contrast but for smooth surfaces (with low roughness) the reduction is relatively small [8].

The changes of roughness (i.e. increase of roughness) of the (0001) plane of cobalt monocrystal resulting from laser induced phase transitions (melting and solidification) and their influence on the structure of magnetic domains observed with SEM are examined. One of the parameters characterizing surface roughness (root mean square roughness σ) was measured with the optical method and He-Ne laser [8].

2. THEORETICAL FOUNDATIONS

2.1. Idea of the σ parameter determination

The main objective of the theory which describes light reflection from rough surfaces is determination of the dependence between the surface roughness parameters and the parameters of the reflected electromagnetic wave [8]. Two methods are usually refered to: perturbation method and Kirchhoff's method. The latter is applied when the linear dimension of irregularities are comparable to the wavelength λ of the applied radiation and any changes of topography are rather smooth. It is comonly assumed that metal surfaces of this kind are produced when conventional, mechanical methods of surface finish are applied (the samples examined here were prepared with such methods).

Kirchhoff's method, also called the tangent plane method, applied for the reflection of electromagnetic wave from rough surfaces, is similar to the Kirchhoff's method of analyzing diffraction phenomenon [12]. The equation for the intensity I_z of speculary reflected light, presented below after the monograph [12] is the result of this method. It was used in this work for the estimation of σ parameter and has the following form:

$$I_z = r(\theta_1) I_o \exp\left[-\frac{16\pi^2\sigma^2\cos^2\theta_1}{\lambda^2}\right] \tag{1}$$

where θ_1 is the angle of incidence, λ is the wavelength of the incident radiation, $r(\theta_1)$ – coefficient or reflection of perfectly smooth surface of the same chemical composition, I_o – the intensity of the incident. More detailed assumptions connected with equation (1) and with the method of determination for (0001) plane of cobalt monocrystal are also presented in [8].

Measurements of I_z for different angles of incidence θ_1 (when λ, I_0, $r(\theta_1)$ are known or it can be assumed that $r(\theta_1)$ = const) make it possible to find σ from equation (1). Its value is given by the tangent of the slope of the line representing the dependence: $\ln I_z = f(\cos^2\theta_1/\lambda^2)$. Such procedure of σ determination is justified when the condition $\sigma < \lambda$ is satisfied, what means that the contribution from diffuse reflection is negligible in the direction of specular reflection.

When the relation between σ and λ is reversed, i.e. $\sigma > \lambda$, what means that diffusive component dominates in the reflected radiation, a characteristic pattern of randomly distributed bright and dark spots, called speckles, can be observed for laser illumination [13]. It is the result of interference of coherent waves reflected from different points of rough surface. Computer analysis of the obtained pattern gives parameters which can be connected with the roughness of the reflecting surface.

2.2. Transport of heat

The general form of the heat conduction equation (for cartesian system of coordinates x-y-z) which must be solved to find the distribution of temperature within the sample (metal) can be written as [14]:

$$\rho_m c_p(T)\frac{\partial T}{\partial t} - \nabla(K(T)\nabla T) = S(x, y, z, t) \tag{2}$$

The first term on the left side of equation (2) includes $\rho_{m.}$ and $c_p(T)$ which represent material's density and specific heat capacity. Both parameters are, in general, temperature dependent. K(T) in the second term stands for thermal conductivity and is temperature dependent, too. The right side S(x,y,z,t) describes the source of laser generated heat and its time evolution. For materials which are thermally anisotropic thermal conductivity K(T) is also direction dependent. In general, the coefficient of thermal conductivity must be represented by tensor with temperature dependent components. All this makes it impossible to solve the equation (2) with analytic methods. As a consequence, the solution of problems connected with heat transport for laser, pulsed heating of anisotropic materials is only realised with numerical methods [15,16].

The problem is even more difficult to tackle when calculations are conducted for cobalt monocrystal which for temperatures below the melting point changes its structure from hexagonal to cubic and at the Curie temperature turns from ferromagnetic to paramagnetic. These circumstances make the dependence $c_p(T)$ complex. One also has to treat the thermal conductivity K(T) in the hexagonal phase as a tensor and as a scalar in the cubic phase.

It is well known that radiation emitted by pulsed laser Nd:YAG (λ_l = 1,06 μm) [17] is absorbed within the superficial layer of the cobalt sample of depth about $l_\lambda = 4\cdot10^{-3}$ μm. Comparison of this depth and the diameter D of the laser beam leads to relation $D \gg \sqrt{\alpha\tau} \gg l_\lambda$, where $\alpha = 0{,}22\cdot10^{-4}$ m^2/s stands for thermal diffusivity of the cobalt monocrystal in the direction parallel to the c axis (perpendicular to the (0001) plane) and $\tau = 5\cdot10^{-9}$ s is the duration of the laser pulse. It is clear now that the source of heat resulting in this method can be treated as a surface source. It is also assumed in this paper that the laser pulse has rectangular time shape, the examined material is isotropic and its properties do not depend on temperature. One can then estimate the temperature change in the superficial layer from the following equation [14]:

$$\Delta T(z,t) = 2Q(1-R)\sqrt{\alpha t}K^{-1} ierfc\left[\frac{z}{2\sqrt{\alpha t}}\right] \tag{3}$$

where

$$\alpha = \frac{K}{\rho_m c_p} \tag{4}$$

and $0 < t \le \tau$; Q is the intensity of laser radiation; R is the coefficient of effective reflection; z denotes depth; $ierfc(x)$ – a factor resulting from the error analysis [14].

For $z = 0$ and $t = \tau$ the temperature rise of the surface from Eq. (3) can be obtained as:

$$\Delta T(0,\tau) = 2Q(1-R)K^{-1}\sqrt{\frac{\alpha\tau}{\pi}} \tag{5}$$

2.3. Capillary waves

In the presented examinations the light beam emitted by Nd:YAG laser is focused on the sample's surface with the system of spherical lenses and the intensity of radiation is so adjusted that, when absorbed in the superficial layer, it causes melting but without washing out or rapid evaporation. In the locally created basin of molten metal (when the rest of the surface has the temperature of the surrounding medium) complex physical processes take place during illumination and after the laser pulse is ceased.

Most of the papers on the interaction of laser light with solid samples deal with the topography of rapidly "frozen" surface and the properties of the modified superficial layer [18]. On this basis conclusions are made about possible dynamic phenomena which could occur in the basin of molten metal during the laser pulse and subsequent solidification. Feedback processes between the laser radiation and illuminated object can also be analysed [19-21]. Thorough and laborious microscopic observations (with optical and SEM technique) of the laser produced crater prove that the surface is subject to distortions and axial-symmetric undulation called capillary or ring waves, surface tension waves or ring-like structures [9].

The phenomenon of capillary waves generation has not been fully examined experimentally and described with complete theory, so far [22-26].

But even the present state of understanding of the effect makes plausible the thesis that it can be important in laser technological processes and metallurgy for the following reasons:

a) interaction between laser radiation and oscillating surface of the melted metal can lead to resonance processes (knife-type melting [23]);

b) wave oscillations cause mixing of liquid metal (especially close to the boundary of the melted region) what in a mechanical way enables gas penetration into the superficial layer of metal, thus changing its properties [27].

c) if the radial structures on the solidified surface are regarded as a frozen image of capillary wave, then the phenomenon can be used for measurements of surface tension and viscosity of liquid metals (especially if one could produce capillary waves with linear wave fronts [18, 22, 27]).

d) on the same assumption as above, the minimum density of energy flux required to produce the capillary waves can be regarded as the threshold value of local surface melting of the examined sample; it is especially important in situations when one has to know whether the process of laser modification of superficial layer undergoes from solid or liquid phase [28].

The physical mechanism of capillary waves generated in the above described way has not yet been analysed in full extent. For instance, according to Ruikalin [29]: "the nature of this phenomenon in different melting processes of locally heated metals is considered only in a limited range, while for the melting with laser light pulses it is not examined at all". Ruikalin [29] and Magill [22] claim that ring structures represent the image of radially propagating capillary waves, excited by the rapid increase of vapour pressure in the centre of melted region. Magill [22] also pyromertically recorded periodic [24] temperature changes on the surface (with the period T_0 about 10 μs) upon melting with quasi-stationary (500 μs duration) laser pulse.

The similar effect was independently observed by Chuen-Horng Tsai [30]. These experimental facts let one assume that the observed periodicity of temperature is the source of periodic changes of vapour pressure what creates the driving force of vibrations on the surface of the produced crater. For this reason both Ruikalin [29] and Magill [22] propose to apply classic dispersive formula for capillary waves of wave number k also for this kind of waves [31]:

$$\omega_0{}^2 = \frac{\sigma k^3}{\rho} \; for \; a_c = \sqrt{\frac{2\sigma}{\rho g}} >> \lambda_c \tag{6}$$

where: σ – the coefficient of surface tension, ρ – density of the liquid metal, a_c – the capillarity coefficient.

If the discussed phenomenon proceeds as described above then, according to Magill [22] this equation could be used for measurements of surface tension and viscosity of liquid metals in wide range of temperatures (λ_c can be found from the distance between crests within the crater and the frequency ω_0 from the oscillations of temperature on the surface).

Other possible cause of molten metal oscillations in the centre of the heated region and radial propagation of this disturbance as the capillary wave can be rapid end of laser heating. This mechanism is quite probable when the heating is realised with laser working in quasi-stationary regime [29]. On the other hand, when the pulsed laser in free-generation mode is used for local heating then few last spikes in the pulse can produce oscillations in the liquid metal basin [27].

Quite different mechanism (so far not confirmed with appropriate experimental evidence) was proposed by Levchenko and Chernyakov [25]. They used linearization of the Navier-Stokes equation to show that upon the heating with high enough intensity (from the free side of the surface film) undumped thermo-capillary waves can occur on the surface of the liquid metal as the result of the action of thermocapillary forces. Propagation of these waves on the surface of molten metal (during laser illumination) can significantly change the conditions of light reflection and absorption. It also has influence on mass and heat transport in the melted region what can be of special importance in laser modification of metals from liquid phase. The description of this mechanism is not complete because of nonlinear initial phase which still needs further examinations.

3. EXPERIMENTAL PROCEDURE

A specimen of cobalt monocrystal, oriented by means of an X-ray diffractometer, was cut from a sample of suitable quality so that the observation plane was perpendicular to the direction of the magnetic anisotropy axis [0001]. This specimen was disc-shaped. Its height (parallel to [0001] axis) was (6.07±0.01) mm; the diameter of the observed surface was (9.04±0.01) mm.

The surface of the examined specimen was smoothed by mechanical polishing. After that, the surface roughness was studied (root mean square roughness σ was measured) by optical method (laser He-Ne, wavelength λ = 0.6328 μm). Schematic diagram of the experimental apparatus used to measure the σ was published elsewhere [8]. Afterwards the picture of the domains visible in the examined plane (due to magnetic contrast of the first type) was registered by SEM (BS300 type). Images were obtained using the electron beam with energy 20 KeV and current 100 pA at normal incidence.

Next, this previously smoothed surface was roughened by melting with pulses of radiation from Nd YAG lasers: model CTL-1501/A (E = 50 mJ, τ = 5 ns, λ_1 = 1.06 µm, nanosecond pulse) and model KWANT 15 (E = 8,0 J, τ = 4.5 ms, λ_1 = 1.06 µm, millisecond pulse). In the area of laser operation local melting of a thin layer of material was observed. At the end the parameter σ was assessed (by optical method) and the pictures of melted areas were registered (by means of SEM).

4. RESULTS AND DISCUSSION

4.1. Assesement of σ

The angular dependence of the intensity of light speculary reflected from the investigated specimen is shown in Fig. 3 [8], where $\ln\frac{r(\theta_1)I_o}{I_z}$ is plotted vs $\frac{16\pi^2\cos^2\theta_1}{\lambda^2}$. In this case the angular dependence of $r(\theta_1)$ is computed from the Fresnel equations for light polarized perpendiculary to the plane defined by incident and reflected beams [12]. It can be seen from Eq.(1) that if the theory of scattering from rough surfaces is applicable, the resulting straight line has a slope m $=\sigma^2$. The roughness of the (0001) plane of the examined sample was characterised with the root mean square roughness σ = 0.19 µm.

4.2. SEM observations

A maze pattern of magnetic contrast typical for cobalt monocrystal, observed by SEM on the surface prepared for mechanical or laser roughening, is presented in Fig. 1. The surface in Fig. 1 (σ = 0.19 µm) is „smooth" and the magnetic contrast is not reduced by the topographic contrast.

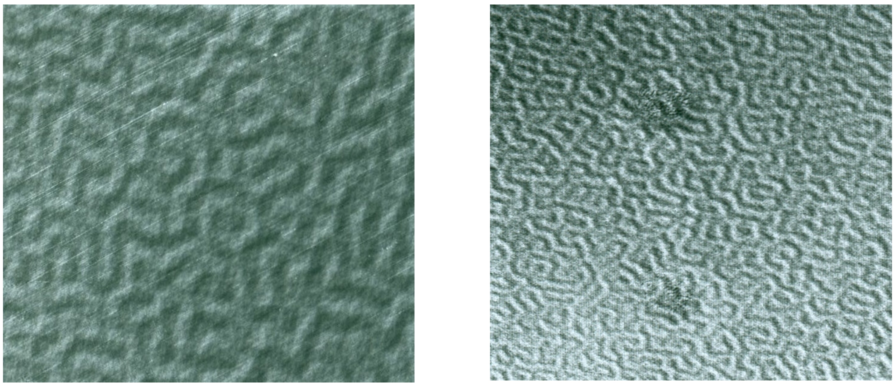

Fig. 1. Maze-like pattern of magnetic contrast observed by SEM on the surface prepared by mechanical polishing (left photo, σ = 0.19 μm). The domains mean width is d ≈ 70 μm. Two darker regions in the central part of the photo are the spots melted with nanosecond pulse of radiation from Nd:YAG laser

When short pulse (~1ns) of laser radiation is incident on the metal, dramatic effects associated with the laser-induced temperature change can occur. These effects may include melting of the thin superficial layer. If the laser pulse is switched off, the melted region is rapidly cooled. In rapidly-frozen areas (after melting with laser pulse radiation) different kinds of waves were noticed [9,18].

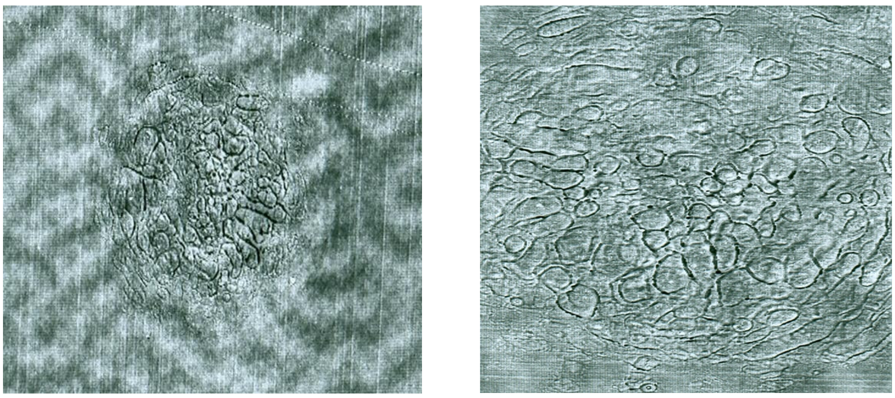

Fig. 2. Laser roughened surface of the sample. Different magnifications of the same melted area are presented

In this paper the nonlinear capillary waves are observed (Fig. 2). Their presence means that the superficial layer of the exposed area was molten and its temperature was not lower than $T_{mpt} = 1766$ K. Moreover, their presence means that surface roughness increased (in molten area). The parameter σ of this surface is to high to be assessed (measured) by optical method, used in this investigations. Therefore, one could only make and assumption that $\sigma > 0.52$ μm [8]. In spite of this the type-I magnetic contrast is not reduced. Plausibility of the assumption that the parameter σ satisfies the condition: $\sigma > \lambda$ is justified by the fact that laser speckles are observed in the direction of specular reflection (Fig. 3).

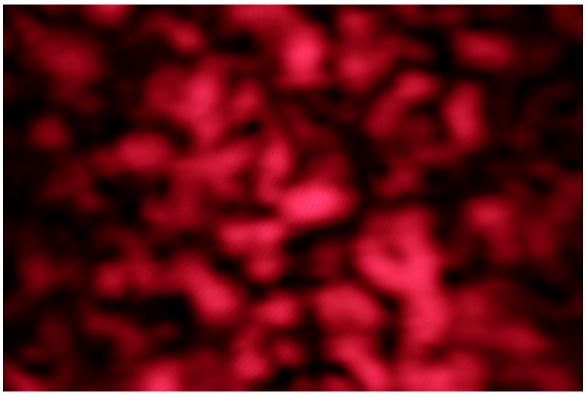

Fig. 3. Laser speckles observed in the direction of specular reflection from the region melted with nanosecond laser pulse

4.3. Calculations of the temperature profile

For the calculations of the temperature profile (temperature vs. depth) of the area heated with nanosecond pulse, the Eq. (3) and necessary parameters, listed in Table 1, were used. This temperature profile is shown in Fig. 4. We can see (from Fig. 4) that the melting temperature (T_{mpt}) was achieved on the depth $z_{mpt} \approx 150$ nm, the Curie temperature T_c was reached on the depth $z_c \approx 250$ nm and finally the martensite transformation (hcp too fcc phase transition) temperature T_m was obtained for the depth $z_m \approx 550$ nm.

It is clear from the analysis of the above presented photographs that melting took place in the region of interaction with laser light but despite this magnetic domains of the Kittel type can be seen. It can mean that the magnetic field normal to the plane (0001), which results because of considerable anisotropy, is sufficiently strong to satisfy the condition of creation of the Kittel type, open magnetic domains.

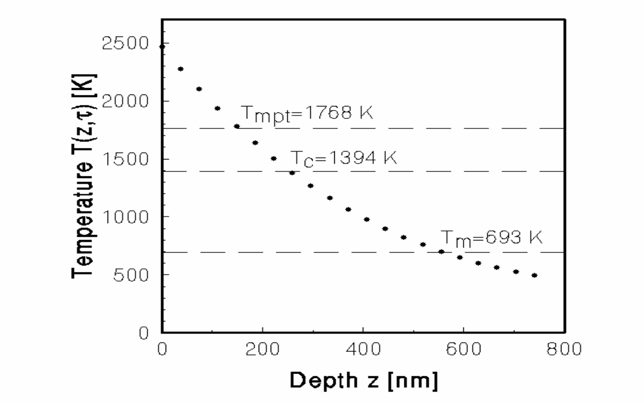

Fig. 4. Temperature vs depth (temperature profile)

Table 1

Some parameters of the sample and experiment data used for the calculations of the temperature profile T(z,t) from Eq. (3)

Solid density ρ_s (kg m^{-3})	8900 [17]
Liquid density ρ_l (kg m^{-3})	7740 [17]
Melting point T_{mpt} (K)	1765 [17]
Temperature of martensite transformation T_m (K)	693 [18]
Laser pulse duration τ(ns)	5
Laser pulse energy E (J)	$50*10^{-3}$
Thermal conductivity K (W m^{-1} K^{-1}) for 273 K	104 [19]
Reflection coefficient R (%)	73 [1]
Diameter of the irradiated region (laser spot) D (m)	$2.5*10^{-3}$
Specific heat c_p (J kg^{-1}K^{-1})	442 [20]
Power density Q (W m^{-2})	$2.0*10^{12}$
Thermal diffusivity α (m^2 s^{-1}), assumed from Eq. (4)	$2.8*10^{-5}$
Temperature rise of the surface $\Delta T(0,\tau)$ (K)	$2.0*10^{3}$

4.4. Capillary waves observed with SEM

Photographs below present the SEM images of the regions of the sample melted with laser pulse of millisecond duration. A series of overlapping regions of this kind can be seen on Fig. 5.

Clearly visible axial-symmetric, radial structures called surface tension waves or capillary waves set ("frozen") on the sample's surface prove that local melting took place. Open magnetic domains of the Kittel type are not observed in the melted spots and close to them. Left photograph in Fig. 5 also proves excellent (100%) repeatability of the effect of the capillary waves generation and characteristic deformation of the irradiated regions after final solidification.

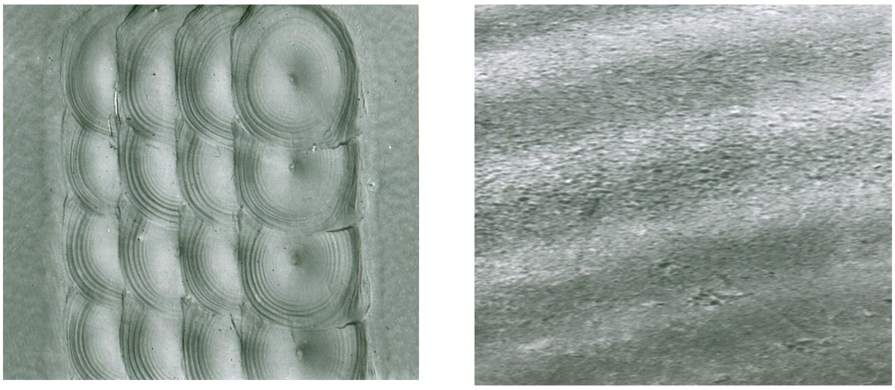

Fig. 5. Photographs of the series of melted regions (left), each with diameter about 400 μm and the magnified section of one crater (right); the waves on this photograph have wavelength of about 25 μm

Very gentle and slow polishing of the melted material which removes the layer of the thickness smaller than 100μm does not reveal the Kittel type magnetic domains (as checked with SEM). Fig. 6 presents the photograph of the boundary of the melted spot (left side of the picture) when the melted layer was removed to the depth of about 60 μm. Before this photograph was made the surface was gently polished, so that the parameter σ (measured with the optical method) was equal to circa 0.19 μm.

The width of the Kittel type, open magnetic domains close to the boundary of the crater is considerably smaller than in the right side of the photograph (Fig. 6), far from the laser melted spot. This observation proves the existence of thermal stresses along the boundary between the melted and unchanged regions.

It was checked that the removal of the surface film slightly thicker than 100 μm finally reveals the Kittel type magnetic domains.

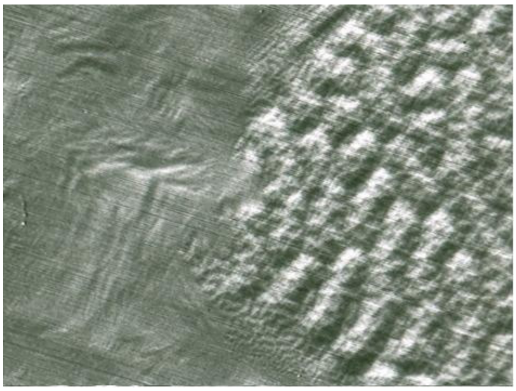

Fig. 6. Photograph of the SEM image of the boundary of the region melted with milisecond laser pulse after the removal of the melted material to the depth of about 60 μm

5. CONCLUSIONS

The roughness of the (0001) plane of the examined sample, before laser melting, was characterised with the root mean square roughness $\sigma = 0.19$ μm and, as a consequence, type-I magnetic contrast in SEM observations was not reduced by topographic contrast. The value of roughness parameter σ was determined optically with He-Ne laser light.

Though the superficial layer of the metal irradiated with laser nanosecond pulse undergoes temporary melting, magnetic domains of the Kittel type can be seen through this layer. It can mean that the magnetic field normal to the plane (0001), which results because of the considerable anisotropy of the metal under the melted spot, is still sufficiently strong to satisfy the condition of creation of the Kittel type, open magnetic domains. Calculations conducted in this paper prove that the thickness of this melted layer is about 180 nm.

When melting is produced with milisecond pulses open magnetic domains of the Kittel type are not observed in the melted spots and close to them. In this case the magnetic domains close to the boundary of the crater are considerably smaller than these which are far from. This effect, called refining of the domains, prooves the existence of thermal stresses along the boundary betwepen the melted and unchanged regions. On the surface of the regions melted with milisecond pulses capillary waves can be observed.

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LASEROWE TOPNIENIE POWIERZCHNIOWE MONOKRYSZTAŁU KOBALTU

Streszczenie

W pracy przedstawiono opis zjawiska laserowego topnienia powierzchniowego monokryształu kobaltu oraz zjawisk z nim związanych. W badaniach stosowano nano i milisekundowe impulsy promieniowana elektromagnetycznego, emitowanego przez lasery Nd:YAG. Promieniowanie kierowano na powierzchnię (0001) monokryształu kobaltu. Zbadano wpływ chropowatości powierzchni (0001) na obraz domen magnetycznych (I-y typ magnetycznego kontrastu) monokryształu kobaltu, obserwowanych za pomocą SEM. Chropowatość powierzchni zmieniano stosując metodę polerowania z użyciem diamentowych proszków i topnienie powierzchniowe nanosekundowym impulsem laserowej energii. Średnie kwadratowe odchylenie profilu σ badanych powierzchni oszacowano refleksometryczną metodą optyczną. Obliczono głębokość obszaru stopionego nanosekundowym impulsem laserowym, stosując izotropowy model badanego metalu oraz niezależność stałych materiałowych od temperatury. Przeprowadzono jakościową dyskusję zjawisk (generacja fal kapilarnych oraz rafinacja domen magnetycznych typu Kittela) powstających podczas powierzchniowego topnienia warstwy wierzchniej monokryształu kobaltu impulsem milisekundowym.