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N.B.: When citing this work, cite the original article.

Original Publication:

Antonella Lorusso, F Gontad, Esteban Broitman, E Chiadroni and Walter Perrone, Characterisation of Pb thin films prepared by the nanosecond pulsed laser deposition technique for photocathode application, 2015, Thin Solid Films, (579), 50-56.

<http://dx.doi.org/10.1016/j.tsf.2015.02.033>

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Postprint available at: Linköping University Electronic Press

<http://urn.kb.se/resolve?urn=urn:nbn:se:liu:diva-115145>

Characterization of Pb thin films prepared by the ns pulsed laser deposition technique for photocathode application

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ABSTRACT

Pb thin films were prepared by the ns pulsed laser deposition technique on Si (100) and polycrystalline Nb substrates for photocathode application. As the photoemission performances of a cathode are strongly affected by its surface characteristics, the Pb films were grown at different substrate temperatures with the aim of modifying the morphology and structure of thin films. Atomic force microscopy and scanning electron microscopy analyses showed a strong morphological change in the deposited films with the substrate temperature, and the formation of spherical grains at higher temperatures with the nucleation of large voids on the film surface. X-ray diffraction measurements showed that a preferred orientation of Pb (111) normal to the substrate was achieved at 30 °C while the Pb (200) plane became strongly pronounced with the increase in the substrate temperature. Finally, a Pb thin film deposited on Nb substrate at 30 °C and tested as the photocathode showed interesting results for the application of such a device in superconducting radio-frequency guns.

Keywords: Pb thin films, pulsed laser deposition, metallic photocathodes

I. INTRODUCTION

The deposition of thin films with adequate morphology and a crystalline structure is a key point in the development of many research fields. During the last two decades, the pulsed laser deposition (PLD) technique has been applied increasingly to the synthesis of thin films because of its versatility for the deposition of practically any kind of material with a relatively simple experimental set-up [1, 2]. The versatility of PLD also lies in the possibility of obtaining films very adherent to the substrates, even at room temperature and with a high predictable growth rate, which can be precisely controlled through a priori studies of the experimental parameters. Adequate selection of the irradiation conditions, as well as the chemical and physical properties of the target materials, is crucial for the production of very adherent thin films [3–5]. Moreover, the choice of substrate temperature may also affect the quality of thin films from the morphological and structural features point of view [6].

In this paper, we report the characterization of Pb thin films deposited by PLD grown on Si (100) and on polycrystalline Nb substrates at different temperatures with a laser wavelength of 266 nm. The deposition process of Pb thin films by PLD technique was also studied by using the fundamental wavelength of Nd:YAG at 1064 nm [7]. However, the deposited films were non-homogeneous with a high droplet density on the film surface. It is well known that the quality of the deposited films is strongly related to the laser parameters, such as laser wavelength and laser fluence. In the same paper was also showed that the droplet density lowered by the laser fluence. Therefore, in this work the laser fluence was fixed as closely as possible to the ablation threshold in order to reduce

the thermal effects on the target during the ablation process decreasing, in this way, the formation of the melted material which is responsible of the presence of droplets on the film surface. The substrate temperature was changed as effort to improve the homogeneity of the Pb thin films which is very important for the application of such device as photocathode.

The study is of great interest for the R&D of photocathodes and in particular for Nb superconducting radio-frequency guns (SRF), which combine the advantages of photo-assisted production of high brightness and short electron pulses with reduced electrical losses and continuous wave operation [8, 9]. SRF cavities present a main drawback in the low quantum efficiency (QE) of the material used for their fabrication ($QE_{Nb} \sim 2 \times 10^{-5}$ @ 250 nm) with respect to other metallic photocathodes, reducing the possibility of obtaining electron beams of high current [10].

The most promising alternative seems to be the insertion of a small photo-emitting spot made of an alternative material, which improves the photoemission performance of the SRF cavity but preserves its quality factor [11]. The use of Pb has been proposed as an excellent solution because its superconducting critical temperature of 7.2 K is quite similar to that of Nb (9.3 K) and the QE of Pb is an order of magnitude higher than that of Nb [10].

With the idea of a hybrid Nb/Pb cathode, after a dedicated study to find the most suitable experimental conditions to get Pb thin films with morphological and structural characteristics adequate for a photocathode, we deposited a Pb thin film on a Nb substrate to test the photoemission performances of such device comparing the results with a Pb bulk cathode.

II. EXPERIMENTAL SET-UP

All the films were deposited by focusing the fourth harmonic of a Q-switched Nd:YAG laser (266 nm, Continuum Powerlite-8010, $\tau = 7$ ns, $f = 10$ Hz) on the target surface, which was placed in a high vacuum system. The working laser fluence chosen was close to the laser ablation threshold of Pb, F_{thr} , in order to reduce the laser thermal effect on the target as much as possible. F_{thr} was computed according to equation (1) [7]:

$$F_{thr} = \frac{\rho L_T}{3} \left(\frac{c_s \Delta T + \Delta H_f + \Delta H_e}{1 - R} \right) \quad (1)$$

where ρ is the material density, $L_T = \sqrt{2D\tau} = 0.6 \mu\text{m}$ is the thermal diffusion length, D is the thermal diffusivity, τ is the laser pulse duration, c_s is the specific heat, ΔT is the difference between the melting point of Pb, T_m , and the room temperature, ΔH_f is the latent heat of fusion, ΔH_e is the latent heat of evaporation and R is the surface reflectivity of the Pb target. The parameters are reported in Table 1. Equation (1) is always valid for ns laser ablation of metals where the optical penetration depth of the laser is much less than L_T .

The adequate laser fluence was selected by decreasing the laser beam energy with an attenuator and it was fixed at about 0.5 J/cm^2 , very close to the theoretical $F_{thr} = 0.4 \text{ J/cm}^2$, calculated considering that $R = 0.5$ at 266 nm in Eq. (1). This value was found empirically by observing the worsening of the vacuum down to 5×10^{-5} Pa during the irradiation

process. Mass spectrometry investigations were also carried out to follow the quality of the vacuum. Such method, even if not accurate, is very fast and versatile to get an idea of the range of the laser ablation threshold and enough to reduce the thermal effects on the target.

A detailed description of the experimental apparatus is described elsewhere [12], while the experimental conditions for the PLD thin film deposition are reported in Table 2. The films were grown at different substrate temperatures, ranging from 30 to 230 °C, by using an ohmic heater system. The Si (100) substrates were used as-received without any additional surface polishing treatment, while the Nb substrate was ultrasonically cleaned in acetone for 30 min and dried by high purity dry nitrogen gas.

The average ablation rate was 0.27 ± 0.02 $\mu\text{g/pulse}$, deduced by weighing the target before and after the ablation process, which indicated a target surface etching rate of 16 nm/pulse, namely 8×10^{14} atoms/pulse.

The characterization of the as-deposited Pb thin film morphology was undertaken by scanning electron microscopy (SEM, model JEOL-JSM-6480LV) operating at 20 kV of electron accelerating voltage and atomic force microscopy (AFM, Nanoscope III controller with Digital Instruments Multimode head, integrated with J-scanner) in tapping mode.

The structure and crystal orientation of the material was studied by Cu K_{α} ($\lambda = 1.5405$ Å) X-ray diffraction (XRD) in $\theta/2\theta$ mode by using a PANalytical X'Pert-PRO Materials Research Diffractometer.

The adhesion of the Pb films to the Nb substrate was evaluated by the Daimler-Benz Rockwell-C (DBRC) adhesion test method [13]. Indentation tests were carried out with a

standard Rockwell hardness tester fitted with a Rockwell-C-type diamond cone indenter with an applied load of 150 kg. The adhesion result is obtained by using an optical microscope and classifying the amount and length of radial crack lines and the delamination and/or buckling features by different levels, which determine the adhesion strength from HF level 1 to 6 according to the VDI 3198 German standard [13]. HF1 shows excellent adhesion properties with a few crack networks while HF6 shows the poorest adhesion properties with complete de-lamination of the film.

Finally, the QE of the films was measured in a home-made photodiode cell [14]. The vacuum chamber, in which the photocathode was inserted, was evacuated at a base pressure of about 2×10^{-6} Pa by means of ionic and turbomolecular pumps. The quality of the vacuum was controlled by a quadrupole mass spectrometer. The photocathode drive laser ($\lambda = 266$ nm) was the same as that used in the PLD experiments. The energy density on the cathode was controlled by adjustment of both the mask size and the telescopic focusing lens. The anode consisted of a copper ring of 25 mm in diameter separated from the photocathode at a distance of 3 mm. The anode was biased at DC voltages up to 5 kV thus allowing the generation inside the gap of a maximum electric field of about 1.7 MV/m.

III. RESULTS AND DISCUSSION

A. Pb FILMS DEPOSITED ON Si SUBSTRATES

Preliminary depositions were carried out on Si substrates at different temperatures in order to optimize the experimental conditions.

Figure 1 shows the AFM images of Pb thin films deposited at substrate temperatures of 30, 90, 160, and 230 °C. At room temperature the film presents a quite contiguous morphology (Fig. 1a) while the increment of the substrate temperature favours the formation of non-wetting clusters (Figs. 1 b–d). This behaviour is in accordance with Warrender and Aziz's model [15, 16] concerning the growth of metal-on-insulator thin films: at the beginning of the deposition thin films typically grow according to the Volmer–Weber mode, in which atoms grow in three-dimensional islands on the surface [17, 18]. As the islands grow larger, they start to impinge each other driven by capillarity forces inducing the formation of clusters. Further deposition joins these elongated clusters, forming a tortuous network of island chains with the presence of holes and voids till a quite contiguous film with further deposition is formed. In this model the substrate temperature, T , is a key parameter in the growth process of the thin film because the time scale, t , for the formation of such elongated and interconnected clusters is $t \propto T$ which means that the increment of the substrate temperature induces a delay in the formation of a contiguous film as confirmed by our experimental results. Moreover, at the highest substrate temperatures (Figs. 1 c and d), the film growth is characterized by the formation of nanometric spherical islands, which tend to increase in height, while their cross section decreases, with the temperature. In fact, the estimated average cross-section diameters of

the islands were about 350 nm at 160 °C and 270 nm at 230 °C, while their height was increased, as can clearly be seen on the three-dimensional AFM images. This effect could be caused by the creation of adatom-vacancy pairs, which provokes the motion of the atoms upwards with the morphological transition from larger and shorter to thinner and taller islands inducing a steep increment of the film Root Mean Square (RMS) roughness after 160°C of the substrate temperature as shown in Fig. 2.

XRD patterns of Pb thin films at different substrate temperatures are reported in Fig. 3. Several Pb peaks are ascribed to (111), (200), (220) and (311) planes of cubic Pb, correspondingly [19]. The polycrystallinity of the films is attributed to the effect of energetic species present in the plasma plume, which promote the atom mobility at the substrate surface. Time of flight measurements demonstrated, indeed, that the ion mean kinetic energy of the plasma can reach some hundreds of eV depending on the laser fluence [20, 21]. The appearance of a few weak diffraction peaks located at 27.68°, 38.12° and 44.04°, indicated with an asterisk (*) in the d pattern of Fig. 3, could be provoked by the formation of lead silicates (most likely PbSiO₃) during the first steps of the deposition process [22, 23]. The interaction of energetic Pb atoms and ions with both physisorbed molecular oxygen and the native oxide layer during the first stage of PLD could lead to the formation of lead silicate crystallites.

However, the most interesting feature of the XRD pattern of the deposited films is the strong evolution of relative peak intensities of Pb (111) and Pb (200) with the substrate temperature. At 30 °C (a pattern of Fig. 3), the contribution of the (111) crystalline planes of the Pb network is more pronounced with respect to the others, showing a preferential orientation along those planes typical of polycrystalline metallic thin films. Nevertheless,

as the substrate temperature increases, the relative intensity of Pb (200) with respect to Pb (111) becomes greater and greater (*b-d* patterns of Fig. 3) showing a sort of epitaxial growth of the Pb film which follows the crystalline orientation of the Si (100) substrate. Moreover, at 230°C the Si (200) peak, ascribed to the Si substrate, is evident because, as discussed above, the film grows in the shape of thin and tall islands with the formation of large voids and holes as depicted in Fig. 4 a. The Si (200) contribution is not evident in the case of the film grown at 30 °C, because its morphology is characterized by interconnected and elongated clusters, which improve the substrate coverage (see Fig. 4b). Many micrometre droplets of different sizes are evident on the film surface of Fig. 4 derived from the ejection of melting material directly from the Pb target during the ablation process because, even if the working laser fluence of 0.5 J/cm² is close to the ablation threshold (0.4 J/cm²), the melting of the target cannot be ruled out. The maximum surface temperature of the target can be calculated by equation (2):

$$T_s = 2I_0(1 - R)\tau^{1/2} / (\pi\kappa\rho c_s)^{1/2} \quad (2)$$

where $I_0=100$ MW/cm² is the working laser power density, κ is the thermal conductivity of Pb and the laser pulse was considered with a rectangular temporal distribution [24]. The value of about 750 K, higher than the boiling point of Pb (600 K), was obtained ignoring the plasma absorption of the laser pulse with our experimental conditions [25]. The crystallite size, S , related to the Pb (111) and Pb (200) peaks at 230°C has been calculated by the Debye–Scherrer formula: $S = k\lambda / (b \cos \theta)$ [26], where $k=1.11$ is the Debye–Scherrer constant, λ is the Cu_{K α} wavelength (1.5405 Å), b is the full-width at high maximum (FWHM) of the peak and θ is the Bragg angle for the corresponding peak. b is the average of two values obtained by considering two Gaussian curves in the Pb (111)

(Fig. 5 a) and Pb (200) peaks (Fig. 5 b) for the $\text{CuK}_{\alpha 1}$ and $\text{CuK}_{\alpha 2}$ weighted contributions. The average value obtained from the two FWHMs of the two Gaussian curves, subtracting the XRD instrumental broadening, gives the b value useful in the Debye–Scherrer formula. The resulting crystallite sizes are 120 and 180 nm for Pb (111) and Pb (200), respectively.

B. Pb FILM DEPOSITED ON Nb SUBSTRATE

The above results showed that the substrate temperature is an interesting experimental parameter in the development of a Pb cathode based on thin film with well-organized nanostructure grains and controlled epitaxial grown by increasing the substrate temperature. Such features, in fact, could improve the photoemission performances of the cathode by the field emission effect [27-29] but the formation of large voids and holes on the film surface limits the application of such a device as a cathode. Further studies will be required in the future to improve the morphology of cathodes based on nanostructured Pb thin film.

After this study concerning the role of the substrate temperature on the Pb film growth, a sample of Pb film on Nb substrate was prepared to be installed in the photodiode cell to test it as a photocathode fixing the Nb substrate temperature at 30 °C . The film thickness was of about 300 nm deduced by analysing in cross section the film deposited on the Si substrate in the same experimental conditions (Fig. 6a). Figure 6b shows the SEM image of the Pb target track produced by laser ablation at 0.5 J/cm² and after 15,000 laser pulses (700 pulses/site). The thermal effect, such as melting, is evident, as is the formation of asperities and depressions.

The film deposited on polycrystalline Nb substrate was characterized by a structure and morphology similar to that deposited on Si substrate. Moreover, the deposited Pb film was extremely adherent to the Nb substrates, as the scotch and the DBRC adhesion tests revealed. In particular, the DBRC adhesion test showed no visible delaminations around the indentation crater and the presence of very few cracks, typical signs of the optimal adhesion strength quality *HFI*.

In this configuration we studied the photoemission performances of the Pb film and, for comparison, of the Pb bulk (see open square data in Fig. 7). The low *QE* value of about 3×10^{-5} was associated with the desorption of contaminants due to the exposure of the photocathodes in the open air before the installation in the photodiode cell. For this reason, in situ laser cleaning treatment was applied with 6000 laser shots @10 Hz of repetition rate and a laser energy density of about 40 mJ/cm^2 which was sufficient to remove the contamination compounds from the cathode surface but well below to the laser ablation threshold of the Pb cathode (400 mJ/cm^2). The black square data of Fig. 7 show the photoemission performance of thin film and bulk after the laser cleaning treatment. Nonetheless, the relationship between the collected charge and the number of photons arriving on the cathode surface was linear only up to a total charge of about 250 pC. Above that threshold, space charge effect influenced the measurements of the electron charge. According to this effect, experimental data were located under the straight line obtained by the fit of data in the low charge limit. The linear trend indicated that the photoelectron emission process occurred mainly via the one-photon absorption mechanism, as predicted by the generalised Fowler–Dubridge equation:

$$J = CI^n \quad (3)$$

where J is the current density, C is a constant, I is the laser intensity and n is the number of photons absorbed per emitted electron [30, 31].

The corresponding value of QE for the film and the bulk before and after the laser cleaning is reported in Fig. 8 and it was calculated as: $\frac{N_e}{N_\phi(1-R)}$ where N_e is the number

of the photoemitted electrons and N_ϕ is the number of photons which arrived on the cathode surface taking into account the Pb reflectivity. QE for the photocathode based on Pb thin film was around 8×10^{-5} with a reduction of the value till 6×10^{-5} due to the space charge effect (Fig. 8a). Before the laser cleaning treatment QE was almost 3×10^{-5} . The QE values of the photocathode based on Pb bulk were around 6×10^{-5} and 2×10^{-5} after and before the laser cleaning treatment, respectively (Fig. 8b). We suppose that the improvement of the QE value for the photocathode based on thin film could be attributed, in same way, to the interconnected grain morphology of the film.

IV. CONCLUSIONS

Pb thin films were grown on Si (100) substrates at different substrate temperature with the aim of optimizing the experimental deposition conditions. All deposited films were characterized by SEM, AFM and XRD analyses revealing that the substrate temperature is an interesting parameter to modify the morphology and structure of the Pb films. Nevertheless, the nucleation of large voids at higher temperatures limits the application of such devices as photocathodes. The Pb film grown on Nb polycrystalline substrate at

30°C was used to deduce the photoemission properties of the sample. The film was very adherent showing interconnected island morphology and a polycrystalline structure similar to that deposited on Si substrate. The QE test of Pb photocathode based on thin film, after the in-situ laser cleaning processing, gives a value higher than that of Pb bulk.

ACKNOWLEDGMENTS

This work was supported by the Italian National Institute of Nuclear Physics (INFN) and partially funded by the Italian Minister of Research in the framework of FIRB – Fondo per gli Investimenti della Ricerca di Base, Project no. RBFR12NK5K. The authors are very grateful to Dr. L. Persano for AFM measurements and to V. Tasco for XRD measurements. E. Broitman acknowledges the Swedish Government Strategic Research Area in Materials Science on Functional Materials at Linköping University (Faculty Grant SFO-Mat-LiU # 2009-00971).

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FIGURE AND TABLE CAPTIONS

Table 1. Chemical and physical properties of Pb.

Table 2. Experimental conditions for Pb film deposition.

Figure 1. 3D AFM images of Pb thin films grown at different substrate temperatures: (a) 30 °C, (b) 90 °C, (c) 160 °C and (d) 230 °C.

Figure 2. RMS roughness of Pb thin films obtained at different substrate temperatures. The continuous line is a guide to the eye.

Figure 3. XRD patterns of the polycrystalline Pb thin films at different substrate temperatures: (a) 30 °C, (b) 90 °C, (c) 160 °C and (d) 230 °C. The peaks indicated by an asterisk (*) could be ascribed to lead silicates.

Figure 4. SEM images of Pb film on Si (100) substrate at a) 230°C and b) 30°C.

Figure 5. Deconvolution of a) Pb (111) and b) Pb (200) by two Gaussian curves (dash curves). The crystallite sizes were deduced by the Debye–Scherrer formula resulting in 120 and 180 nm for Pb (111) and Pb (200), respectively.

Figure 6. (a) Cross section of Pb thin film on Si (100) substrate at 30°C and (b) the SEM image of the Pb target track produced by laser ablation at 0.5 J/cm² and after 15,000 laser pulses (650 pulses/site).

Figure 7. Charge emitted from a) Pb film and b) Pb bulk before (□) and after (■) laser cleaning. Continuous lines are the data-fitting curves taking into account the equation (3).

Figure 8. QE of a) Pb film and b) Pb bulk before ($\square$) and after ($\blacksquare$) laser cleaning as a function of the laser energy.

Table 1

Parameter	Pb
Thermal conductivity κ ($\text{W}\cdot\text{cm}^{-1}\cdot\text{K}^{-1}$)	0.35
Thermal diffusivity D ($\text{cm}^2\cdot\text{s}^{-1}$)	0.24
Specific heat c_s ($\text{J}\cdot\text{g}^{-1}\cdot\text{K}^{-1}$)	0.13
Mass density ρ ($\text{g}\cdot\text{cm}^{-3}$)	11.43
Melting point T_m (K)	600.6
Boiling point T_b (K)	2022.0
Latent heat of fusion ΔH_f ($\text{J}\cdot\text{g}^{-1}$)	24.5
Latent heat of evaporation ΔH_e ($\text{J}\cdot\text{g}^{-1}$)	860

Table 2

Target	Pb
Substrate	Si (100) at 30, 90, 160, 230°C, Nb polycrystalline at 30 °C
Target–substrate distance	4 cm
Laser spot size	1.2 mm
Laser pulse duration	7 ns
Laser fluence	0.5 J/cm ²
Number of pulses for target cleaning before deposition	3,000
Number of pulses during deposition	15,000
Pulses/site	700
Background pressure	6×10 ⁻⁶ Pa

FIGURE 1

FIGURE 2

FIGURE 3

FIGURE 4

FIGURE 5

FIGURE 6

FIGURE 7

FIGURE 8