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Influence of laser energy density on the plasma expansion dynamics and film stoichiometry during laser ablation of BiSrCaCuO

J. Gonzalo and C. N. Afonso^{a)}

Instituto de Optica, CSIC, Serrano 121, 28006 Madrid, Spain

J. Perrière

Groupe de Physique des Solides, Universités Paris VII et VI, URA 17, Tour 23, 2 place Jussieu, 75251 Paris Cedex 05, France

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The plasma expansion dynamics and the composition of films deposited during laser ablation of BiSrCaCuO at laser energy densities in a broad interval ($1\text{--}6\text{ J/cm}^2$) have been studied by means of spatially resolved real time optical emission spectroscopy and Rutherford backscattering and nuclear reactive analysis, respectively. In vacuum, the velocity of the ejected species is found to increase as the energy density increases whereas the compositional and angular distribution of the deposited films remain unchanged. When an oxygen pressure of 10^{-1} mbar is applied, the film composition and angular distribution depend on the laser energy density. The results are analyzed in the frame of a collisional mechanism between the ejected and gas species, the effect of increasing the laser energy density being similar to that of decreasing the oxygen pressure. Finally, this competitive influence of the oxygen pressure and the energy density is discussed within the adiabatic expansion model. © 1996 American Institute of Physics. [S0021-8979(96)03610-X]

I. INTRODUCTION

In the last few years, pulsed laser deposition has become a field of increasing importance in the growth of high-quality thin films. A wide variety of materials such as high- T_c superconductors, ferroelectrics, metals, semiconductors, or organic compounds has been successfully grown by this technique.^{1,2} However, in spite of advantages such as conservation of the stoichiometry, compatibility with a reactive atmosphere or low-temperature substrate requirements, among others, the quality of the deposited films is extremely sensitive to the experimental parameters used during the deposition process. Films with the desired properties are thus only obtained in a limited range of substrate temperature, gas pressure, and laser energy density. Therefore, a precise knowledge of the influence of the controllable experimental parameters on the plasma expansion and deposition process is essential to optimize the widespread use of this technique. While the substrate temperature mainly affects to the amorphous or crystalline character of the deposited films,^{3,4} the laser energy density together with the gas pressure determines the microstructure, the composition, and lateral distribution of the deposited films.³⁻⁶

The influence of a gas pressure during the deposition of high- T_c superconductors has been widely studied; the plasma expansion dynamics changes from a free expansion in vacuum to a collision-dominated one in the presence of a gas background.⁷⁻¹⁰ The gas atoms and/or molecules collide with the species present in the plasma, slowing down their velocity. Depending on the gas pressure, shock or drag models have been used to describe the plasma expansion process.^{7,8,11,12} As a consequence, the film stoichiometry and material angular distribution are strongly dependent on both the gas pressure and nature.^{5,6,8,13} On the other hand, al-

though the material removal process is actually known to be dependent of the laser energy density,^{4,14} its influence on the deposition process and film composition is not yet well understood. Several works have pointed out that the plasma characteristics such as temperature, degree of ionization, electron density, nature, and kinetic energy and distribution of the species present depend on the laser energy density.^{7,11,14-18} Particularly, the increase of the kinetic energy of the plasma species influences the structure and/or composition of the growing film; either through bombardment effects,¹⁹ or through preferential resputtering of some of the elements from the substrate as in the case of ferroelectrics deposited in vacuum.²⁰ These results thus suggest the existence of a strong correlation between the kinetic of the plasma species and the material distribution in the deposited films. The aim of this work is then to study this correlation through the analysis of the influence of the laser energy density both on the plasma expansion dynamics and the film stoichiometry.

II. EXPERIMENT

Laser ablation of a rotating BiSrCaCuO target is performed by using an ArF excimer laser [$\lambda=193\text{ nm}$, pulse duration: 12 ns full width at half-maximum (FWHM)] at an incidence angle of 45° . The target is placed in a vacuum chamber equipped with a multitarget holder and evacuated to a residual pressure of 4×10^{-8} mbar. Experiments in vacuum and at 10^{-1} mbar of oxygen pressure are performed at laser energy densities up to 6 J/cm^2 . The films were grown on Si wafers (100) held at room temperature and located at 33 mm from the target surface. 2212 and 4334 targets are used for the plasma studies and since no significant differences are observed on the plasma emission, the films are only grown from the 2212 target. Nuclear microanalysis, by the direct observation of nuclear reactions and backscattered particles,

^{a)}Electronic mail: cnafonso@pinar1.csic.es

TABLE I. Transitions and energies of the upper and lower levels responsible for the emission lines studied in this work.

Species	Wavelength	Transition	E_{upper} (eV)	E_{lower} (eV)
Ca ⁺	396.85	$4p\ ^2P_{1/2} \rightarrow 4s\ ^2S_{1/2}$	3.12	0
Sr ⁺	416.18	$6s\ ^2S_{1/2} \rightarrow 5p\ ^2P_{1/2}$	5.91	2.98
Sr ⁺	421.55	$5p\ ^2P_{1/2} \rightarrow 5s\ ^2S_{1/2}$	2.98	0
Ca	422.68	$4p\ ^1P_1 \rightarrow 4s\ ^1S_0$	2.93	0
Ca	428.30	$4p\ ^2P_2 \rightarrow 4p\ ^3P_1$	4.78	1.89
Sr	460.73	$5p\ ^1P_1 \rightarrow 5s\ ^2S_{1/2}$	2.69	0
Bi	472.22	$7s\ ^4P_{1/2} \rightarrow 6p\ ^3D_{3/2}$	4.04	1.42
Sr	474.19	$5p\ ^2P_{1/2} \rightarrow 5p\ ^3P_0^0$	4.41	1.80
Cu	510.55	$4p\ ^2P_{3/2} \rightarrow 4s\ ^2D_{5/2}$	3.82	1.39

is used to study the homogeneity and composition of the deposited films. The ¹⁶O content is measured through the ¹⁶O (*d,p*) ¹⁷O* nuclear reaction using a 0.85 MeV deuteron beam, absolute values being obtained within 3%. The cation content in the films is obtained by Rutherford backscattering spectrometry using a ⁴He⁺ at 2.2 MeV, absolute values being obtained using the RUMP simulation program.²¹

The plasma produced by the laser ablation process is imaged with a $\times 2$ magnification onto the entrance slit of a spectrometer (SPEX, 0.05 nm resolution). The optical image of the plume is scanned along the normal to the target with a spatial resolution of 60 μm . The emitted light is collected by a photomultiplier (15 ns rise time), connected to a 500 MHz digitizer for transient emission measurements. Further details of the experimental setup can be found elsewhere.^{6,9}

III. RESULTS

A. Plasma emission

The emission spectrum recorded in the 390–600 nm range at 2 mm from the target surface shows no emission from excited oxidized diatomic molecules both in vacuum and at 10^{-1} mbar of O₂. The observed emission lines correspond to ions (Ca⁺, Sr⁺) and neutrals (Bi*, Sr*, Ca*, Cu*) and most of them could be identified according to standard tabulations.²² The emission lines studied in this work are summarized in Table I, and since it has been shown that the behavior of the emission lines depends on the excitation energy of the electronic transition responsible of the emission,⁹ we have selected three calcium emission lines to show the most representative results: Lines at 396.85 nm (Ca⁺) and 422.68 nm (Ca*) with excitation energies ~ 3 eV and line at 428.30 nm (Ca*) with an excitation energy ~ 5 eV. Further details of the analysis of the emission spectra can be found elsewhere.⁹

Figure 1 shows the dependence of the transient emission intensity maximum I_M on the laser energy density during ablation in vacuum for the three considered transitions at a distance $d=1.7$ mm to the target surface. This distance is selected since the emission from the continuum has completely disappeared and the emission from the different species is close to its maximum value.⁹ No emission could be detected from any species for energy densities below 0.3 J/cm², whereas two different regions are observed for higher values. The emission intensity shows a sharp increase for

FIG. 1. Maximum emission intensity I_M as a function of the laser energy density from (■) Ca⁺* (396.85 nm), (○) Ca* (422.68 nm), and (△) Ca* (428.30 nm) emission lines. Results are recorded in vacuum at $d=1.7$ mm from the target surface.

energies in the range 0.3–1.5 J/cm², to increase more slowly for higher energy densities. As is shown in Fig. 1, the relative increase of the emission is dependent on the particular emission line: It is two times higher for the emission lines at 396.85 nm (Ca⁺*) and 422.68 nm (Ca*) than for the 428.30 nm line (Ca*). A similar dependence of I_M on the laser energy density is found in the presence of 10^{-1} mbar of oxygen.

From the values of the delay between the laser pulse and the transient emission intensity maximum t_M , obtained at $d=1.7$ mm at different laser energy densities, it is not straightforward to estimate the expansion velocities of the ejected species because different effects contribute to t_M and some of them may be energy dependent: the time necessary to have promoted species into the excited state, the lifetime of the excited state, or the time the species need to travel from $d=0$ to $d=1.7$ mm. Since the last contribution is the only one related to the expansion velocity of the ejected species, the evolution of t_M with the distance d to the target surface [$t_M=f(d)$] has to be studied for each laser energy density. In vacuum, this dependence is linear and therefore the most probable velocity of the species can be estimated from the inverse of the slope of this linear dependence.⁹ Figure 2 shows the dependence of the expansion velocities on the laser energy density calculated for the three calcium transitions studied. The values are in all cases of the same order of magnitude and they increase as the laser energy density increases following a similar function. Since the observed dependence of t_M on distance in 10^{-1} mbar of oxygen is similar to that obtained in vacuum for distances to the target $d \leq 12$ mm,⁹ the most probable velocity should be similar to that in vacuum and, therefore, a similar dependence on the laser energy density is expected in 10^{-1} mbar of oxygen.

B. Film stoichiometry

Films were deposited at different laser energy densities in the range 1–6 J/cm² both in vacuum (4×10^{-8} mbar) and in oxygen (10^{-1} mbar). This pressure is selected since it has been previously reported to lead to stoichiometric films.¹³ Figure 3 shows the number of deposited atoms determined

FIG. 2. Expansion velocities as a function of the laser energy density for (■) Ca^{+*} (396.85 nm), (○) Ca^* (422.68 nm), and (△) Ca^* (428.30 nm) emission lines. The values are determined from the inverse of the slope of the spatially resolved spectroscopic measurements.

from Rutherford backscattering spectroscopy (RBS) and nuclear reactive analysis (NRA) along the film direction contained in the incidence plane of the laser beam (X axis) and normalized to their maximum value ($X=0$), for Bi, Sr, Ca, Cu, and O. In vacuum, all the species show a similar angular distribution even for films grown at different laser energy densities; therefore, the average value is the only one plotted (dashed line) in Figs. 3(a) and 3(b). The presence of an oxygen atmosphere produces not only a broadening of the angular distribution, but also a distribution which depends on the mass of the element.⁶ The distribution is narrower for the heaviest species (Bi) and broader for lighter species (Ca and O). When the laser energy density is increased in the presence of an oxygen atmosphere, this broadening decreases as can be seen by comparing the distributions measured in films grown at 1 J/cm^2 [Fig. 3(a)] and 2.8 J/cm^2 [Fig. 3(b)].

The results summarized in Fig. 3 suggest that the film composition of films grown in oxygen should be dependent

FIG. 3. Lateral distribution of the elements [(●) Bi, (■) Sr, (△) Ca, (◇) Cu, and (□) O] along the X axis contained in the incidence plane of the laser beam for films deposited in 10^{-1} mbar of oxygen at (a) 1.0 J/cm^2 and (b) 2.8 J/cm^2 . The average distribution obtained in films deposited in vacuum is plotted as a dashed line for comparison. The number of atoms of each element has been normalized to its value at $X=0$. The inset shows the film deposition geometry.

FIG. 4. Normalized relative content $[(N_x/N_{\text{cat}})_{\text{ox}}/(N_x/N_{\text{cat}})_{\text{vac}}]$ of films grown in 10^{-1} mbar of oxygen (ox) to those grown in vacuum (vac) at different laser densities. The relative content is measured in the maximum of the angular distribution ($X=0$). The value 1.0 in the Y axis corresponds to the relative content of films grown in vacuum.

on the laser energy density as opposed to that of films grown in vacuum, which exhibit always the same composition. Figure 4 shows the dependence of the relative content of each cation on the laser energy density for films grown in oxygen. The plotted values are measured at the maximum of the distribution ($X=0$) and they have been normalized to the value measured in vacuum, i.e., $(N_x/N_{\text{cat}})_{\text{ox}}/(N_x/N_{\text{cat}})_{\text{vac}}$, where $x=\text{Bi, Sr, Ca, and Cu}$ and $N_{\text{cat}}=N_{\text{Bi}}+N_{\text{Sr}}+N_{\text{Ca}}+N_{\text{Cu}}$. The results show that the composition of the films grown in oxygen tends to that of the films grown in vacuum when the laser energy density increases: The excess of Bi decreases and the Ca and Sr deficiencies tend to disappear. Finally, the Cu relative content, which is always similar to that measured in vacuum, shows no appreciable variation when increasing the laser energy density.

IV. DISCUSSION

The present results show that the emission intensity of the excited species increases as the energy density is increased as shown in Fig. 1. A similar dependence was found when studying the etch rate of YBaCuO targets for different energy densities,^{23,24} thus suggesting that the amount of material ejected from the target increases as the energy density increases. These results allow one to conclude that the increase of the emission intensity is related to an increase of the density of the excited species in the plasma. Nevertheless, this density tends to saturate for energy densities higher than 1.5 J/cm^2 which indicates that the excitation mechanism becomes less efficient for high-energy densities. At high energy densities, the partial absorption of the laser energy by the ejected species through an inverse Bremsstrahlung process becomes important.^{15,16} Then, the amount of laser energy which reaches the target increases at a lower rate and therefore the increase rate of the density of excited species becomes also smaller. Furthermore, the comparison of the evolution of the intensity for lines with a low (~ 3 eV) and high (~ 5 eV) excitation energies shows clearly that the increase of the emission intensity depends on the excitation

energy of the considered emission line, rather than on the ionized character of the species as we had suggested in an earlier work.²⁵

The results presented in Fig. 2 show clearly that the velocity of the ejected species increases as the energy density is increased, this result being obtained during ablation in vacuum. Similar results have been reported during ablation of YBaCaCuO from ion current measurements^{26,27} or emission spectroscopy^{17,18} and they are in agreement with the models proposed to explain the plasma expansion: isothermal expansion followed by an adiabatic expansion¹⁵ or isentropic supersonic expansion.¹⁷ Since the plasma expansion in vacuum has been modelled as a free expansion, the kinetic energy of the species arriving at the substrate should increase with the laser energy density. The presence of energetic species has been reported to affect to the composition of the deposited films because of the existence of resputtering processes.²⁰ Nevertheless, our results show that the increase of the ejected species velocity has no significant influence on the angular distribution of the deposited species or the film composition when deposition takes place in vacuum (Fig. 3); therefore, resputtering processes should be independent on the laser energy density, at least in the studied interval, and are most likely negligible as has been discussed in an earlier work.²⁸

The presence of an oxygen pressure during the ablation process has dramatic consequences on the angular distribution and composition of the deposited films (Figs. 3 and 4). The broadening of the angular distribution has been earlier related to scattering processes of the ejected species by the atoms or molecules of the oxygen gas.⁶ The present results show that this broadening decreases as the energy density increases. The velocities plotted in Fig. 2 are measured at distances $d \leq 12$ mm from the target. At larger distances, it is expected a decrease of the velocity due to scattering processes in 10^{-1} mbar of O_2 , this decrease being proportional to the initial velocity. In the frame of a collisional scheme, the mean free path λ between collisions is energy dependent [$\lambda = f(E^{1/2})$],¹⁰ and the angular dispersion of the ejected species is related to the number of scattering events that occur along the target–substrate distance $D_{\text{target-substrate}}$. The increase of λ when increasing the laser energy density should then result in a decrease of the number of scattering events and, therefore, in an angular distribution and composition closer to that obtained in vacuum, as experimentally observed (Figs. 3 and 4).

The present results show that the laser energy density and the presence of a gas pressure have opposite effects on the plasma expansion process. Assuming an adiabatic expansion model,^{8,12,26} the ablated material pushes the gas species until the plasma and gas pressures equilibrate. This distance L_p is called the plasma length and, for larger distances, the ejected species are transported to the substrate by diffusion. The size of the plasma is given by⁸

$$L_p = A[(\gamma - 1)E]^{1/3} P_0^{-1/3} \gamma V_i^{(\gamma-1)/3\gamma}, \quad (1)$$

where A is a geometric factor related to the shape of the laser spot at the target surface, γ is the specific heat ratio (C_p/C_v), E is the laser energy density, P_0 is the gas pressure, and V_i is

the initial volume of the plasma ($V_i \approx v_0 \tau_{\text{laser}}$ laser spot size, v_0 being the initial species velocity and τ_{laser} the laser pulse duration).

From Eq. (1), the ratio E/P_0 is the parameter controlling the length of the plasma if the experimental geometry remains constant; therefore, E and P_0 have opposite effects as experimentally observed in the present work. According to this model, the length of the plasma L_p is expected thus to increase as the laser energy density is increased and the composition of films grown in oxygen should be closer to that of films grown in vacuum for a distance $L_p \approx D_{\text{target-substrate}}$.^{4,5,8} Considering the experimental parameters used in this work (spot size = 0.7 mm², τ_{laser} = 12 ns, $v_0 = 1-2 \times 10^6$ cm/s, $\gamma = 1.4$, and $A = 1.6$), the lengths of the plasma in the presence of 10^{-1} mbar of oxygen and for 1.0, 1.9, and 2.8 J/cm² energy densities are 26, 31, and 34 mm respectively. Since the $D_{\text{target-substrate}}$ is in our case 33 mm, the films grown at 2.8 J/cm² are the only ones for which $L_p \approx D_{\text{target-substrate}}$. Figure 4 shows that the composition of these films is the closest to that of the films grown in vacuum, as expected. For lower energy densities $D_{\text{target-substrate}} \approx 1.3L_p$ and $1.1L_p$, therefore, the scattering processes become important and they produce not only the broadening of the distribution but also significant composition deviations.

V. CONCLUSIONS

The increase of the laser energy density during ablation of BiSrCaCuO produces an increase of the kinetic energy and density of the excited species present in the plasma. The comparison of the composition and angular distribution of films grown in vacuum and 10^{-1} mbar of oxygen at increasing energy densities show that the energy density and the presence of a gas have opposed effects in the composition and angular distribution. The increase (decrease) of the relative Bi (Ca/Sr) content when films are grown in oxygen in respect to those grown in vacuum tends to be negligible as the energy density is increased, this behavior being understood in the frame of the adiabatic model for the plasma expansion. It is then concluded that stoichiometric films can be grown in the presence of an oxygen pressure by selecting the appropriate energy density, i.e., an energy density for which the length of the plasma is comparable to the target–substrate distance.

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