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Formation of AlSi10Mg surfaces via selective laser melting: scanning electron microscopy and Raman spectroscopy study

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Abstract. Scanning electron microscopy analysis of surfaces produced via the selective laser melting technique by microsecond laser pulses of AlSi10Mg powders in vacuum and argon has been carried out. Because of such modification the structures are formed with micro- and nanopores with concentration depending on the geometric parameters of the particles in the starting powders, as well as on the atmosphere in which the treatment was performed. For the first time by means of the Raman spectroscopy a considerable change in the mechanical stresses and fraction of crystalline state silicon in the studied structures before and after selective laser melting was registered.

1. Introduction

Nowadays aluminum-based micro- and nanostructures are widely used as metasurfaces in optics and photovoltaics [1–3]. Aluminum is enough promising for a following implementation because of this material is sufficiently cheaper than gold and silver which are also used to produce the plasmonic structures. Usually, different lithographic techniques are involved in technological processes of metasurface formation from aluminum. We offer to discuss and study a possibility of surface fabrication via the selective laser melting (SLM) from aluminum powders. Such technology allows to efficiently fabricate not only 2D but also 3D structures. Also, this approach is almost wasteless technology because of the unsintered powder can be used repeatedly.

However, a set of problems exists to fabricate the appropriate plasmonic structures by means of the SLM: (i) such technological features of the process as the laser beam diameter and powder component size at the microscale does not allow to form the nanorelief and, therefore, the plasmon excitation is highly improbable in the visible spectral range; (ii) to avoid SLM-sample cracking it is preferably to use the aluminum powders doped by Si, while Mg and Cu are beneficial in sense of strengthening [4, 5], as a result the additional studies of optical and structural properties of the melted products are needed; and (iii) also, it is necessary to find ways to reduce the number of pores and roughness, which are inherent to the SLM, on the formed surfaces. The problem (i) can be solved by the large-area [6] metasurface fabrication for the infrared and THz ranges. Two other tasks demand the further technological and searching efforts.
In our work we have studied SLM-surfaces produced in vacuum and argon (Ar) from the widely used AlSi10Mg powders [5, 7] with different micromorphology. The main emphasis was done to the analysis of data obtained by the scanning electron microscopy (SEM) and Raman scattering spectroscopy techniques.

2. Materials and methods

Three types of the commercial AlSi10Mg powders named as A, B and C were used in the SLM. The difference between them is the distinction of particle size and form. Typical SEM images (Carl Zeiss LEO SUPRA 50VP) of the initial powders are shown in figure 1. The form of the particles in A and C powders is almost spheroidal. In contrast to, B powder contains the elongated elements also. The particle size is over the range of 2–35, 7–120 and 25–150 µm for powders A, B and C respectively.

![Figure 1. SEM images of the powders A (a), B (a) and C (c).](image1)

The SLM-process was implemented using a Solar LS LQ529 laser (1064 nm, 150 µs, 46 mJ, 10 Hz), vacuum chamber and two-coordinate automated positioner. The laser beam was focused into the screeed powder. The motion of the vacuum chamber by means of the positioner in a horizontal plane allowed the formation of monolayers 10x10 mm of the melted AlSi10Mg alloy from the initial powder. The vacuum chamber was evacuated until $2 \times 10^{-5}$ bar or filled with Ar under the atmospheric pressure.

To study Raman scattering a Raman spectrometer Horiba Jobin Yvon HR 800 was employed and pumped by a He-Ne laser at the wavelength 632 nm.

3. Results and discussion

Each of the SLM powder was melted at the same focusing and energy in vacuum and argon. The obtained plain structures were named as A-SLM-V, B-SLM-V, C-SLM-V, A-SLM-Ar, B-SLM-Ar and C-SLM-Ar. The first letter in such marking corresponds to the initial powder, SLM means the realized selective laser melting, V and Ar are the vacuum and argon atmosphere used at the processing respectively. Typical SEM images of C-SLM-V sample are shown in figure 2 as an example. It is worth to note the presence of pores with the typical size on the order of tens and hundreds of micrometers (figure 2(a)), as well as the ones with the size ~ 200 nm (figure 2(b)). Most likely, the first mode of pores is defined by the initial powder packing density, while the second one – by solidification features of the eutectic Al-Si melt [7].

![Figure 2. SEM image of the sample C-SLM-V at low (a) and high (b) magnifications.](image2)
All melted samples are the porous solid-state matrices with the different porosity (table 1) which was estimated by quantification of the SEM images with a photo editor. The samples based on A and C powders have smaller porosity in comparison with one for the B-type samples. The main reason is a difference between the packing densities. The sphere-like particles in A and C powders can be arranged closer in contrast to the elongated particles from B powder.

The porosities in the cases of SLM in argon and vacuum also differ. The presence of the buffer gas at a certain pressure causes collisions of their atoms with the melt and, therefore, defines the agglomeration and solidification efficiency under following cooling [8]. In our case this efficiency affects the pore formation. A and B powders are characterized by smaller particle sizes than powder C. Thus, A and B structures have larger surface area in comparison with the last powder. Laser surface induces more effective interactions with the Ar molecules and pore formation in A-SLM-Ar and B-SLM-Ar samples in contrast to A-SLM-V and B-SLM-V fabrication (table 1). On the contrary, there is no sufficient difference between the SLM-samples with large enough grains of C-type in Ar and vacuum.

| Sample       | Porosity (%) | Amorphous silicon fraction (%) |
|--------------|--------------|--------------------------------|
| A-SLM-V      | 28           | 1.2                            |
| B-SLM-V      | 36           | 0.9                            |
| C-SLM-V      | 33           | 5.3                            |
| A-SLM-Ar     | 37           | 16.1                           |
| B-SLM-Ar     | 51           | 8.5                            |
| C-SLM-Ar     | 24           | 10.1                           |

It is necessary to mention that the obtained porosity values are typical for SLM experiments as reported in [4, 5]. From the point of view of metasurface fabrication it is desirable to reduce porosity as much as possible even if the plasmon wavelength exceeds a typical pore size.

Another question concerns possible phase modifications in AlSi10Mg alloy. The Si fraction in such alloy is about 10% [4, 5]. The different phase modifications of Si have an influence not only on such mechanical properties as the tensile strength and durability but also on electrical properties, for example, the conductivity. The last one affects the plasmon excitation efficiency. To study Si phase transformations under SLM treatment the Raman spectroscopy technique proved itself. Typical spectra for a starting and melted powder are shown in figure 3(a) and figure 3(b) respectively.

![Raman spectra](image)

**Figure 3.** Raman spectra of A powder (a) and A-SLM-Ar sample (b). All spectra are normalized to the signal maximum. The dashed straight in part (a) corresponds to the line 520 cm$^{-1}$ for monocrystalline Si. The dashed and dotted curves in part (b) are approximations of the Raman lines for amorphous and monocrystalline Si respectively.
All powders prior to SLM are characterized by a Raman line near 520 cm⁻¹ which corresponds to monocristalline Si. However, this line is shifted to a lower frequency (red shift) toward 512 cm⁻¹ (figure 3(a)). Such behavior can be explained by a tensile stress and corresponding lattice deformation [9] which are consequences of the Al alloy powders fabrication by means of the “atomization of melt” technique.

The Raman spectra for the melted powders look quite different (figure 3(b)). The red shift for the line 520 cm⁻¹ disappears. So, the stresses are negligible after the SLM treatment. Instead a broad line 480 cm⁻¹ appears. The nature of this line may be interpreted both as a proper mode of the Si-solid solution in Al-matrix or as the mode of an amorphous silicon. In the both cases the Raman line must be situated at lower frequencies in comparison with the monocristalline Si mode due to transformation of the long-range crystalline order to the short-range one during the SLM. In our work we suggested the amorphous silicon presence. The similar conclusion has been made in the work [10]. To estimate the amorphous phase fraction the ratio between the Raman line intensities at 480 cm⁻¹ and 520 cm⁻¹ was analyzed for all samples [11]. The results are presented in table 1. The amorphous phase fraction lies within the limits from 8% to 16% for the SLM in Ar, while for the modification in vacuum this value does not exceed several percent. The observed difference can be explained by noticeable gas pressure during the SLM when a sufficient number of Ar atoms prevent the uniform Si crystallization due to heat conductivity of the gas. At the same time, the similar mechanism in vacuum is impossible. Also, a correlation between the maximal porosities and minimal amorphous silicon fraction in samples B-SLM-V and B-SLM-Ar in comparison with other samples at the same atmosphere fabrication is observed. Coming from the non-spherical form of the particles in B powder we have supposed that the particle form in the studied powders affects not only the porosity but also the crystallinity of the samples after SLM. In any case, the amorphous silicon concentration in all samples is insufficient to reduce the conductivity of the surface significantly.

In conclusion, the studied AlSi10Mg alloy powders with the different particle sizes and forms treated by the SLM in Ar and vacuum revealed possibility to use them as a base for the surfaces with the porosity and phase content depending on the buffer gas atmospheres and properties of starting powder. The obtained results seem useful and promising for following investigations of the aluminum-based structures fabricated via SLM and their application in plasmonics.

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