Influence of vacuum annealing on the dispersion of chromium nanofilms deposited onto non-metallic materials

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Introduction

Metallization of the surfaces to be soldered with different metals is often used to obtain soldered joints of both metallic and non-metallic parts. This operation is especially important when joining non-metallic materials, in particular ceramics, sapphire, silicon nitride, etc. Chrome coatings of various thicknesses are often used as metallization of both metallic and non-metallic materials.

For soldering ceramics and other non-metallic materials to each other and to metals, the use of chromium in the form of thin films, in particular nanofilms, is promising.

Cr nanofilms deposited on non-metallic inorganic materials are of primary importance in the processes of joining non-metals (ceramics, glass, single crystals, carbon materials). Metallization of the surfaces of such materials ensures sufficient wetting of them with molten metals, which determines the very technological possibility of forming a soldered joint, and also determines the strength and other properties of the joints.

Considerable interest is caused by thin films of chromium, which are widely used in various fields of technology, in particular in the form of coatings on both metallic and non-metallic surfaces. Thanks to the metallization of the surfaces of non-metallic materials with this metal, it becomes possible to further connect such metallized surfaces by soldering, during which the metal films can be heated to high temperatures. The purpose of this work is to study the kinetics of dispersion-coagulation of chromium nanofilms 100 nm thickness, deposited onto oxide (Al_2O_3, sapphire, quartz glass) and non-oxide (Si_3N_4, glassy carbon) materials and annealed in vacuum at temperatures of 1000-1200 ^oC and different holding times at these temperatures.

Materials and Experimental Procedure

In this paper an electron-beam method for sputtering of Cr films was used.

Solid non-metallic substrates were made of quartz glass, sapphire, ceramics based on Al_2O_3 and Si_3N_4, glassy carbon as small thin plates 4 x 3 x 2 mm in size. One of the flat surfaces of each specimen was well polished to a roughness R_z=0.03 ÷ 0.05 μm. After polishing, all specimens were thoroughly defatted and burned in air at 1100 °C for one hour. Chromium film 100 nm thickness was applied to the annealed surfaces of the samples by electron beam sputtering for 10 minutes. In the process of applying the films, the samples were heated to no more than 100 °C, and the thickness of the coating was measured with a special quartz resonator, which was located in the vacuum chamber of the electron beam unit with the ELU-2 sample next to it. The quality of all applied nanofilms was controlled using a XJL-17 metallographic microscope. The specimens with deposited onto them metal films were annealed in a vacuum chamber for various periods of time (from 5 up to 20 min) and at different temperatures (from 1000 ^oC up to 1200 ^oC) in the vacuum not less than 2 x 10^{-3} Pa.

Annealed specimens were investigated using SEM and AFM microscopy with microphotographs storing. Using these microphotographs, the areas of metal islets on the surface of non-metallic samples were determined by the planimetric method. The experimental data obtained were processed in the form of graphs showing the dependence of the surface area of the samples covered with metal films on the annealing parameters (temperature, time).

Results and Discussion

In this work, we study the dispersion kinetics of chromium nanofilms 100 nm thick deposited both onto oxide (alumina ceramics, sapphire, quartz glass) and non-oxide (silicon nitride ceramics and glassy carbon) materials and annealed in vacuum at temperatures of 1000-1200 °C with exposure at each temperature during from 2 up to 20 min.

The initial chromium films on all substrates were continuous and practically retained their continuity at annealing temperatures up to 1000 °C. After a long exposure (20 min) at 1000 °C, the films on all substrates begin to decompose. This process is intensified at 1100 °C; as a result of annealing at 1200 °C, chromium films disintegrate completely into separate fragments and drops.

As a result of long exposure at 1100 °C, chromium films on silicon nitride ceramics interact with the substrate material, and this interaction is significantly enhanced with an increase of the annealing temperature up to 1200 °C.

It was established that annealing at 1000 ^oC has little effect on the integrity of nickel and chromium films on all investigated non-metallic materials, even after a twenty-minute exposure at this temperature. Visible signs of dispersion of the films on all these materials appear only after annealing at 1100 ^oC, and as a result of annealing at 1200 ^oC, these films disintegrate completely after a ten- and twenty-minute exposure, and those that were applied to silicon nitride and glass carbon also interact in these conditions with the substrate material.

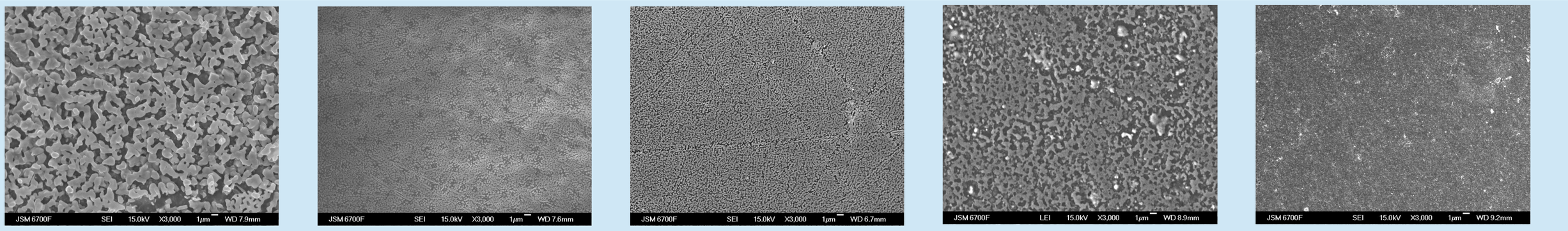

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Fig. 1. SEM images of chromium nanofilm 100 nm thickness deposited onto non-metallic materials and further annealed at temperature 1100 ^oC during 10 min in vacuum: a – quartz glass; b – alumina ceramics; c – leucosapphire; d – silicon nitride ceramics; e – glassy carbon

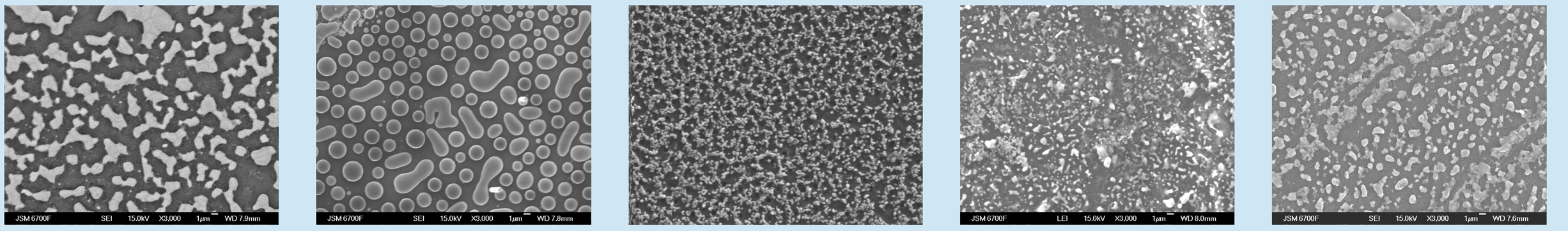

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Fig. 2. SEM images of chromium nanofilm 100 nm thickness deposited onto non-metallic materials and further annealed at temperature 1200 ^oC during 20 min in vacuum: a – quartz glass; b – alumina ceramics; c – leucosapphire; d – silicon nitride ceramics; e – glassy carbon

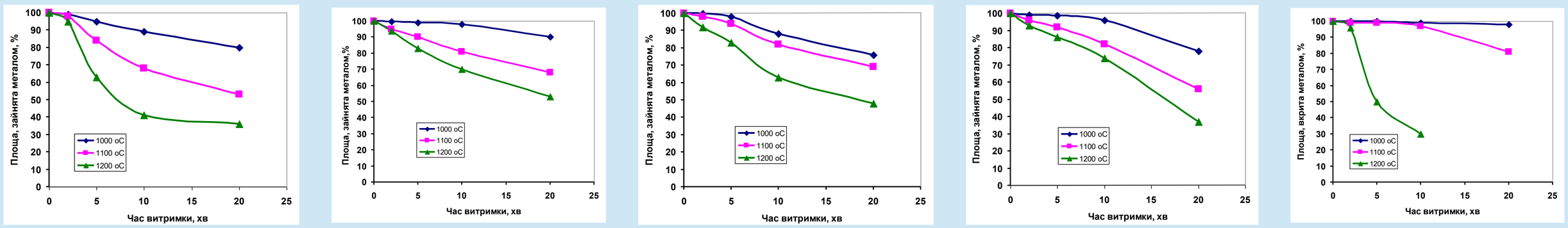

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Fig. 3. Dependence of oxide materials area covered by double niobium-palladium film on annealing time at various temperatures (1000 – 1200 ^oC):a – quartz glass; b – alumina ceramics; c – leucosapphire; d – silicon nitride ceramics; e – glassy carbon

Conclusions

Using the obtained results, it is possible to recommend the use of chromium coatings on all the studied materials for subsequent brazing with appropriate solders at temperatures not exceeding 1100 °C, given that at these temperatures these films do not completely disintegrate, and the non-metallic substrates surface area coated by the metal films is 60-70 % of the original substrates area. At the same time, the units brazing time at 1100 °C should not exceed 10 min.