Influence of Shielding Gas and Mechanical Activation of Metal Powders on the Quality of Surface Sintered Layers

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Abstract. The thesis analyses the influence of argon shielding gas and mechanical activation of PMS-1 copper powder and DSK-F75 cobalt chrome molybdenum powder on the surface sintered layer quality under various sintering conditions. Factors affecting the quality of the sintered surface and internal structure are studied. The obtained results prove positive impact of the shielding gas and mechanical activation. Sintering PMS-1 copper powder in argon shielding gas after mechanical activation leads to reduced internal stresses and roughness, as well as improved strength characteristics of the sintered surface. Analysis of sintered samples of mechanically activated DSK-F75 cobalt chrome molybdenum powder shows that the strength of the sintered surface grows porosity and coagulation changes.

Introduction

Layered synthesis technologies are a rapidly progressing trend in the development of modern industry. A separate line of this development is layered laser sintering of metal powder compositions based on 3D CAD-model allowing applicative products. As far as the layered synthesis of an applicative product basically consists in shaping a single layer, a problem resides in strains which arise in a single sintered layer and thus hamper a uniform application of the next powder layer and deform the product. For a product obtained by laser layered sintering to perform its function, the required quality characteristics must be provided. The main quality parameters of a sintered product are precision, surface layer condition, physical and mathematical properties, durability. A considerable problem in providing quality of the surface layer of a sintered product consists in strains which prevent a uniform application of the following powder layer and distort a product shape. To solve this problem it is necessary to research the influence of physical and mechanical properties of powder materials, laser sintering modes and process conditions of powder layer application [1]. The purpose of this research is to study factors effecting quality and inner structure of a sintered surface as well as to analyze the influence of argon shielding gas, mechanical activation of copper powder PMS-1 and cobalt chrome molybdenum powder DSK-F75 on the quality of a sintered surface layer under different process conditions. Thickness and roughness of a sintered surface and strength of sintered samples have been examined.

Result and Discussion

The sintered process starts with assigning a powder material which directly determines sintering conditions. Important properties of metal powders include their shape, structure, character of interparticle interaction. Particles can be of the following shapes: fibers, flakes, plates, cubes, spheres etc. The size of porous intervals depends on the particle shape and their just proportions. To minimize pores spherical particle are combined with particles of a similar shape and dendritic particles fuse with flaky ones [2]. It also should be kept in mind that powder materials melting temperature decreases as degree of dispersion grows [3]. Packaging density is influenced by particle surface roughness. Mechanical interlocking is one of the types of particle connection [4]. Friction between the particles results from adhesive interaction, that's why great attention is paid to activating powder compositions [5]. When one component powder material is sintered with materials with similar melting temperature under continuous laser impact drops are formed [6]. It should be taken into consideration that thermal physical and chemical properties of powder materials differ from reference ones of solid materials.

Sintering process conditions greatly influence the surface layer quality. Intensity of laser radiation depends on powder material melting temperature, heat conductivity index as well as particle shape and size. For heat-resistant materials laser radiation power must be increased and the speed of laser ray movement must be reduced. When the intensity is improper a powder material is lighted [7].

Increase in powder density leads to decrease in depth of sintering. It can be increased by raising laser radiation power but it causes energy loss.

The following factors considerably influence a prototype quality: coagulation (fusion of molten metal into separate drops), pore formation, internal strains and deformations.

Study of the influence of argon shielding gas on the sintered surface layer quality was carried out with copper powder PMS-1 and cobalt chrome molybdenum powder DSK-F75 under different process conditions. Argon provides a shielding environment which eliminates interaction of powder products with oxygen and nitrogen and also strengthen the product surface. Copper powder PMS-1 being sintered in argon, the sintered surface changed its color, it became goldish, the samples had a harder surface without any cracks [8, 9]. Figures 1-2 shows samples sintered in the air and in argon shielding gas compared. Sintered layer thickness increases from 115mkm up to 200mkm, the sample strength grows.

Figure 1. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.3 mm, t=26 ^{0}C, in the air.

Figure 2. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.3 mm, t=26 ^{0}C, in argon

Figures 3-4 shows samples sintered in argon under the following process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=200 ^{0}C. Cross cracks and longitudinal cracks are not found, sintered layer thickness changes insignificantly from 1675 when sintered in the air to 1735 when sintered in argon.

Figure 3. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=200 ^{0}C, in the air.

Figure 4. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=200 ^{0}C, in argon

Figures 5-6 shows samples sintered under the following conditions P=30 W, V=200 mm/min, S=0.1 mm, t=200 ^{0}C, in the air and in argon. Surface layer quality is dramatically changed. Roughness changes from 525 to 115 mkm, thickness of the sintered layer changes insignificantly from 850 to 915 mkm. The sample sintered in argon lacks cross and longitudinal cracks.

Figure 5. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=200 mm/min, S=0.1 mm, t=200 ^{0}C, in the air.

Figure 6. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=200 mm/min, S=0.1 mm, t=200 ^{0}C, in argon

Sintering in argon shielding gas improves the quality of the surface layer produced of copper powder PMS-1. Roughness is reduced, defects are eliminated.

The influence of mechanical activation on the sintered surface layer quality was studied through sintering copper powder PMS-1 and cobalt chrome molybdenum powder DSK-F75 under different process conditions. Mechanical processing of the powder was carried out in a centrifugal mill AGO-2, in steel mill shells filled with steel balls 6mm in diameter with total mass 600gr and loaded with 30gr of the powder. Mechanical activation increases degree of powder dispersion, aggravates particles crystal lattice imperfection [10] which leads to quick oxidation and allows sintering at unusually low temperatures [11]. Intensive milling of particles increases their sum surface, rises surplus energy of the powder and enlarges surface layer thickness. The mechanical processing was carried out during one, three, five and seven minutes. Figures 4-8 show pictures of sintered cobalt chrome molybdenum surfaces obtained of non-activated and variously activated powders under different processing conditions. In figures 7-11 P=10 W V=100 mm/min, S=0.1 mm t=200 ^{0}C for non-activated sintered surface R_Z=525 mkm, after 1 minute activation Rz=545 mkm after 3 minutes activation Rz=540 mkm, after 5 minutes activation Rz= 615 mkm, coagulation increases, after 7 minutes activation Rz=545 mkm [12].

Figure 7. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=100 mm/min, S=0.1 mm, t=200 ^{0}C, non-activated.

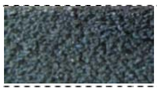

Figure 8. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=100 mm/min, S=0.1 mm, t=200 ^{0}C, after 1 minute activation.

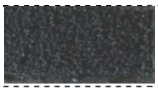

Figure 9. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=100 mm/min, S=0.1 mm, t=200 ^{0}C, 3 minutes activation.

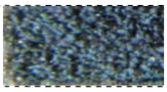

Figure 10. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=100 mm/min, S=0.1 mm, t=200 ^{0}C, after 5 minutes activation.

Figure 11. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=100 mm/min, S=0.1 mm, t=200 ^{0}C, after 7 minutes activation.

Figures 12-16 shows that roughness of the sintered surface changes insignificantly, strength increases. The sintered sample after seven minutes of activation has reduced porosity and solid spots on the surface.

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Figure 12. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=100 mm/min, S=0.1 mm t=200 ^{0}C, non-activated.

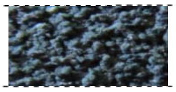

Figure 13. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=100 mm/min, S=0.1 mm t=200 ^{0}C, after 1 minute activation.

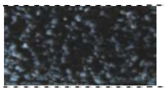

Figure 14. External appearance of sintered surface DSK-F75(x2). Processing modes: P=20 W, V=100 mm/min, S=0.1 mm t=200 ^{0}C, 3 minutes activation.

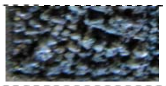

Figure 15. External appearance of sintered surface DSK-F75(x2). Processing modes: P=20 W, V=100 mm/min, S=0.1 mm t=200 ^{0}C, after 5 minutes activation.

Figure 16. External appearance of sintered surface DSK-F75(x2). Processing modes: P=20 W, V=100 mm/min, S=0.1 mm t=200 ^{0}C, after 7 minutes activation.

Increase in activation time leads to reduced diameters of coagulated particles and their uniform distribution on the sintered surface. The strength of the sintered surface increases.

Figure 17. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.1 mm t=26 ^{0}C, non-activated.

Figure 18. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.1 mm t=26 ^{0}C, after 1 minute activation.

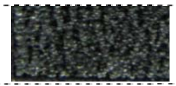

Figure 19. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.1 mm t=26 ^{0}C, 3 minutes activation.

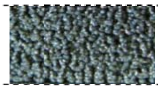

Figure 20. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.1 mm t=26 ^{0}C, after 5 minutes activation.

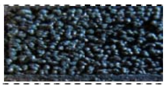

Figure 21. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.1 mm t=26 ^{0}C, after 7 minutes activation.

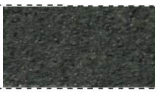

Figure 22. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=300 mm/min, S=0.15 mm, t=26 ^{0}C, non-activated.

Figure 23. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=300 mm/min, S=0.15 mm t=26 ^{0}C, after 1 minute activation.

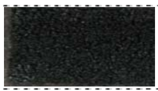

Figure 24. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=300 mm/min, S=0.15 mm, t=26^{0}C, 3 minutes activation.

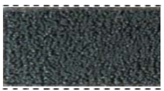

Figure 25. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W V=300 mm/min, S=0.15 mm t=26 ^{0}C, after 5 minutes activation.

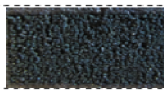

Figure 26. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=10 W, V=300 mm/min, S=0.15mm t=26 ^{0}C, after 7 minutes activation.

Figure 27. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.15 mm, t=26 ^{0}C, non-activated.

Figure 28. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.15 mm, t=26 ^{0}C, after 1 minute activation.

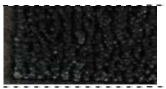

Figure 29. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.15 mm t=26 ^{0}C, 3 minutes activation.

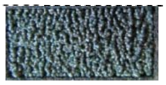

Figure 30. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.15 mm, t=26^{0}C, after 5 minutes activation.

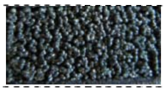

Figure 31. External appearance of sintered surface DSK-F75 (x2). Processing modes: P=20 W, V=300 mm/min, S=0.15 mm, t=26 ^{0}C, after 7 minutes activation.

Figures 32-34 shows diffraction patterns of cobalt chrome molybdenum powder. Y-axis shows diffraction peaks intensity, X-axis shows angular characteristic with maximum shooting angle 2 theta.

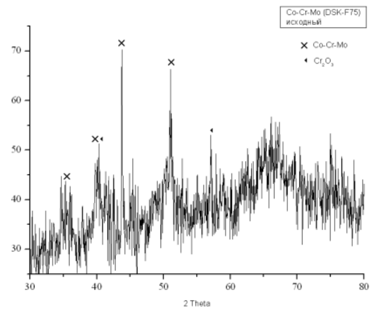

Figure 32. Diffraction pattern of cobalt chrome molybdenum powder, non-activated.

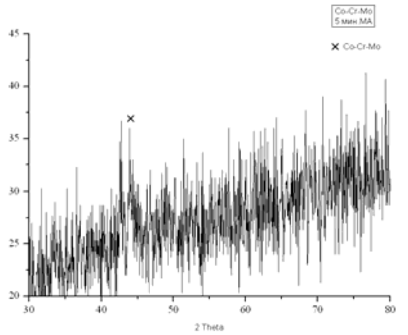

Figure 33. Diffraction pattern of cobalt chrome molybdenum powder, after 5 minutes activation.

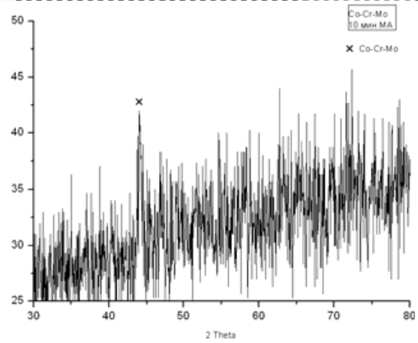

Figure 34. Diffraction pattern of cobalt chrome molybdenum powder, after 10 minutes activation.

Comparison of diffraction patterns of non-activated (Fig.9a) and mechanically activated (Fig.9b) cobalt chrome molybdenum powder shows that after mechanical activation chromic oxide vanishes. Activation time does not considerably influence the diffraction pattern. After activation peaks arise only in one position.

Figures 35-54 show comparative pictures of sintered layers of copper powder composition PMS-1 obtained under different process conditions as well as of non-activated and variously activated powders. Three minutes activation results in increasing strength of a sintered sample, figures 35-37, Rz changes slightly from 625mkm to 630mkm, sintered layer thickness grows from 820mkm up to 950mkm.

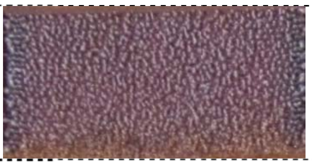

Figure 35. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.3 mm, t=26 ^{0}C, non-activated.

Figure 36. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.3 mm, t=26 ^{0}C, after 1 minute activation

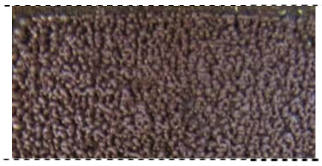

Figure 37. External appearance of PMS-1 surface (x2). Process conditions: P=30 Wt, V=3000 mm/min, S=0.3 mm, t=26 ^{0}C, after 3 minutes activation.

Comparison of two samples figures 38-39 shows improved quality of the surface layer, cracks vanish, roughness is reduced from 975 mkm to 715 mkm.

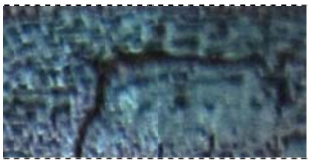

Figure 38. External appearance of sintered surface PMS-1 (x2). Process conditions: P=30 W, V=200 mm/min, S=0.3 mm, t=26 ^{0}C, non-activated.

Figure 39. External appearance of sintered surface PMS-1 (x2). Process conditions: P=30 W, V=200 mm/min, S=0.3 mm, t=26 ^{0}C, after 3 minutes activation.

In figures 40-42 surface roughness of the sintered samples of mechanically activated powder materials decreases from 700mkm to 426mkm.

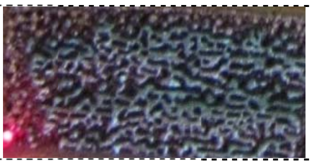

Figure 40. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=26 ^{0}C, non-activated.

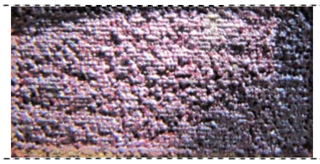

Figure 41. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=26 ^{0}C, after 1 minute activation

Figure 42. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=200 mm/min, S=0.3 mm, t=26 ^{0}C, after 3 minutes activation.

Figure 43. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, non-activated.

Figure 44. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, after 1 minute activation

Figure 45. External appearance of PMS-1 surface (x2). Process conditions: P=30 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, after 3 minutes activation.

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Figure 46. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, non-activated.

Figure 47. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, after 1 minute activation

Figure 48. External appearance of PMS-1 surface (x2). Process conditions: P=15 W, V=3000 mm/min, S=0.1 mm, t=200 ^{0}C, after 3 minutes activation.

Figure 49. External appearance of sintered surface PMS-1 (x2). Process conditions: P=30W, V=200 mm/min, S=0.1 mm, t=26 ^{0}C, non-activated.

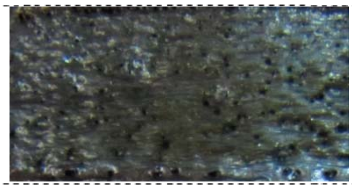

Figure 50. External appearance of sintered surface PMS-1 (x2). Process conditions: P=30 W, V=200 mm/min, S=0.1 mm, t=26 ^{0}C, after 3 minutes activation.

Figure 51. External appearance of sintered surface PMS-1 (x2). Process conditions: P=15 W, V=200 mm/min, S=0.1 mm, t=26 ^{0}C, non-activated.

Figure 52. External appearance of sintered surface PMS-1 (x2). Process conditions: P=15 W, V=200 mm/min, S=0.1 mm, t=26 ^{0}C, after 3 minutes activation.

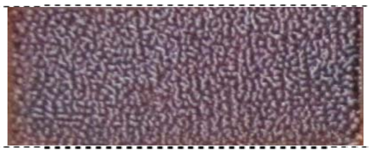

Figure 53. External appearance of sintered surface PMS-1 (x2). Process conditions: P=22 W, V=160 mm/min, S=0.2 mm, t=26 ^{0}C, non-activated.

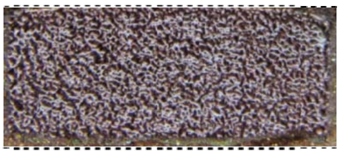

Figure 54. External appearance of sintered surface PMS-1 (x2). Process conditions: P=22 W, V=160 mm/min, S=0.2 mm, t=26 ^{0}C, after 3 minutes activation.

Thus the picture of samples compared clearly show the influence of mechanical activation on the surface quality: it leads to reduced coagulation and roughness.

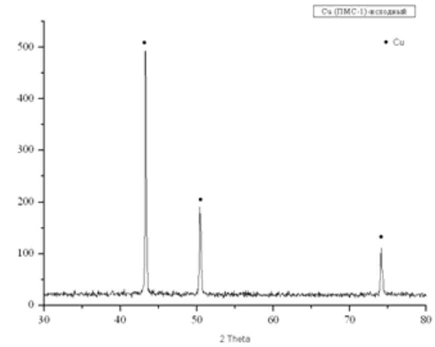

Figure 55. Diffraction pattern of copper powder PMS-1, non-activated.

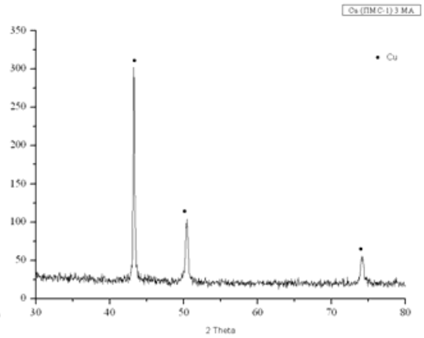

Figure 56. Diffraction pattern of copper powder PMS-1, after 3 minutes activation.

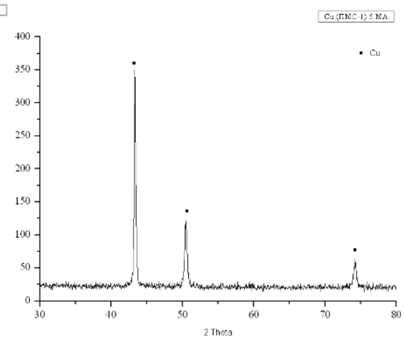

Figure 57. Diffraction pattern of copper powder PMS-1, after 5 minutes activation.

Conclusion

The research in properties of a sintered layer of activated and non-activated powder materials showed that a preliminary mechanical treatment effects the sintering process and provides an improved surface quality: the diameter of coagulated particles decreases, roughness is reduced. Internal structure and strength characteristics are improved.

Positive influence of a shielding gas and mechanical activation of metal powders on the quality of surface sintered layers is proved. In order to reduce roughness and improve the sintered layer quality it is recommended to sinter metal powders subjected to one and three minutes of activation in argon shielding environment which reduces roughness, improves internal structure and strength characteristics.

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