SWAXS Examination of Metallic Alloy Implants Produced by Selective Laser Melting

4. National Crystallography Meeting 16-19 May 2014 Diyarbakir / Turkey

Ahmet Bayırlı[1*], Ilghar Orujalipoor[1], Osman Demir[3] , Ahmet Murat Dursun[3], Semra İde[1,2]

[1]Hacettepe University, Dept. of Nanotechnology and Nanomedicine, 06800, Ankara, Turkey

[3]Hacettepe University, Dept. of Physics Eng., 06800, Ankara, Turkey

[3]Gulhane Military Medical Academy, Medical Design and Manufacturing Center (GATA-METUM), Ankara, Turkey

Abstract

In this research, 12 implant samples have been investigated using small angle X-ray scattering and X-ray diffraction methods. Samples are produced with the SLM method and chosen according to their production angle, annealing temperature and production shape. Aim of this work is to determine external effects of the production media such as annealing temperature, production angle and dimensions. By examining resulting SAXS data, best production parameters will be determined.[1]–[3].

1. Introduction

GATA-METUM (Gülhane Askeri Tıp Akademisi- Medikal Tasarım ve Üretim Merkezi' means Medical Design and Production Center) provides medical implants, prothesis and orthesis for Turkish veterans and citizens. They work with materials like silicon, ceramic or metal. In this study we will be examining Ti6Al4V alloy implants that they produced via SLM technique. We will try to improve nano-structure of the implant via optimizing production parameters.

2.1 SLM

Selective laser melting (SLM) is a 3D printing method which works layer wise. It differs from the selective laser sintering method by using high powered lasers which has enough power to fully melt the material. Laser source selectively scans a powder holder's surface using the map given by a CAD software. When scanning the surface is finished another layer of powder is laid and the whole product is generated repeating these steps. When the end product is generated, it contains place holders as well. Place holders can be separated easily even by its own. [4]. In this study laser melting production parameters such as final angle of the product on table (production angle), final shape of the product and effect annealing temperature are examined. Annealing is a post SLM process to homogenizes the alloy atomic structure by heating.

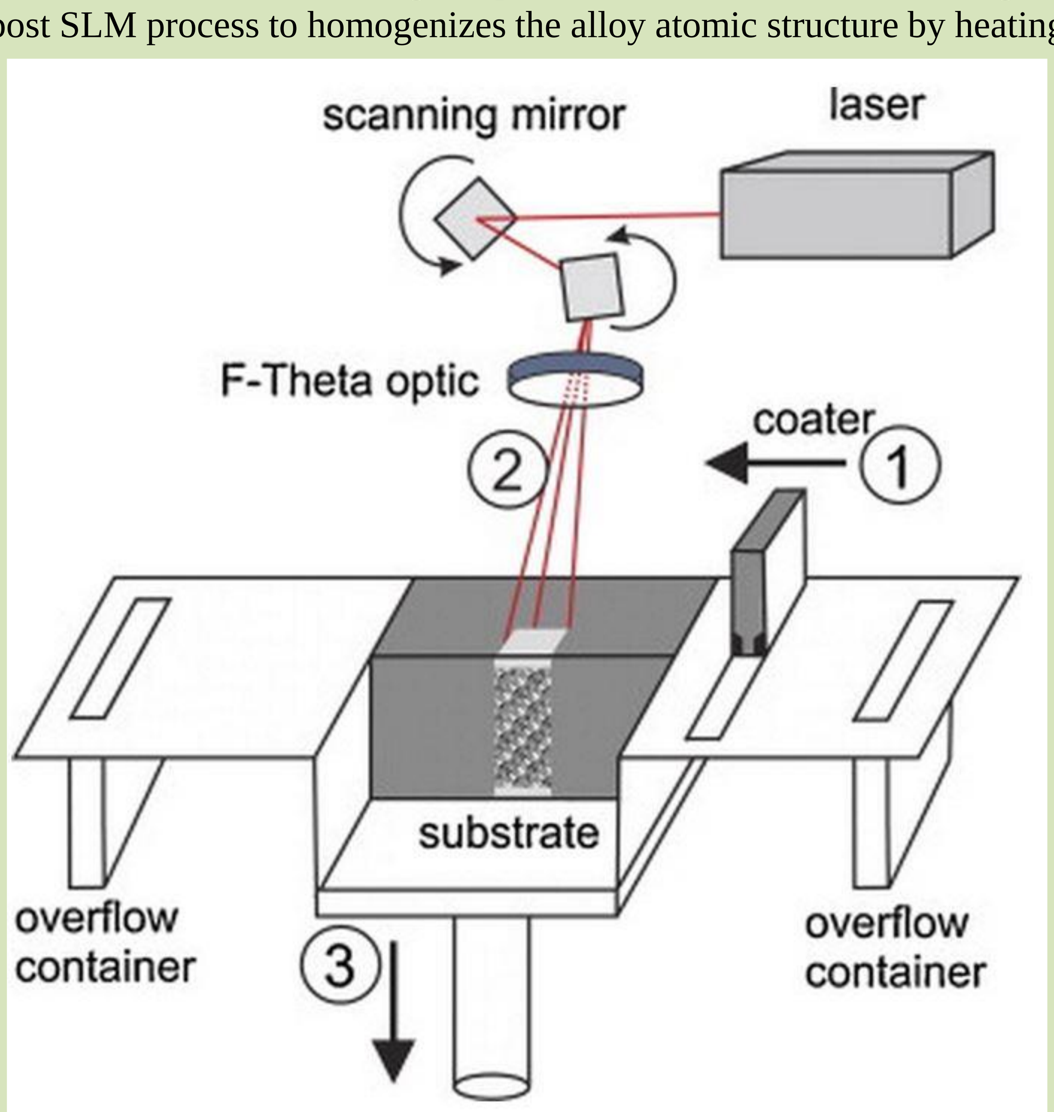


Figure 1. *Schematic diagram of SLM [4]*

2.2 SWAXS Method

Small and wide angle x-ray scattering (SWAXS) experiments were performed with a Kratky compact Hecus (Hecus X-ray systems, Graz, Austria) system equipped with a linear collimation system and X-ray tube Cu target (l= 1.54 Å). The generator was operated at a power of 2 kW (50 kV and 40 mA). Simultaneous measurements of SAXS and WAXS range are possible in the system with two separate linear-position–sensitive detectors used with 1024 channel resolution for each one.

Distance between channels is 54 µm and the distance from sample to detector is 31.5 cm. Scattering curves (SAXS) were monitored and calibrated in q ranges as seen in the Figure 3. All samples were measured for 10 minutes at 23°C by using sample holder of the system.

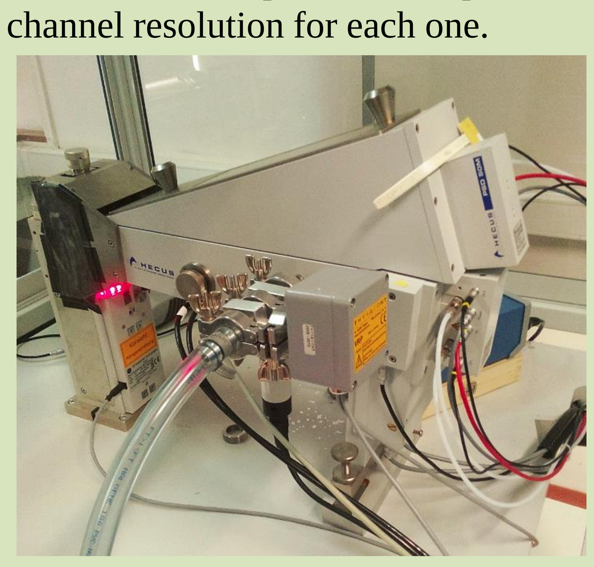


Figure 2. *Hecus SWAXS system in our laboratory*

2.4 Experiments

First experiments are conducted with 12 different samples which are grouped by the production angle, annealing temperature and final shape.

- Group-1 is composed of cylindrical samples as final shape, annealing temperature 840º C and production angle of 0º, 45º and 90º for sample no 1,2 and 3 respectively.
- Group-2 is composed of cylindrical samples as final shape and annealing temperatures 0º C, 840º C and 940º C for sample no 4, 5 and 6 respectively.
- Group-3 is composed of samples 7,8 and 9. Group-3- and group-1- are same except final shape is rectangular prism for group-3-.
- Group-4 is composed of samples 10, 11 and 12. All parameters are same as Group-2- except final shape is rectangular prism.

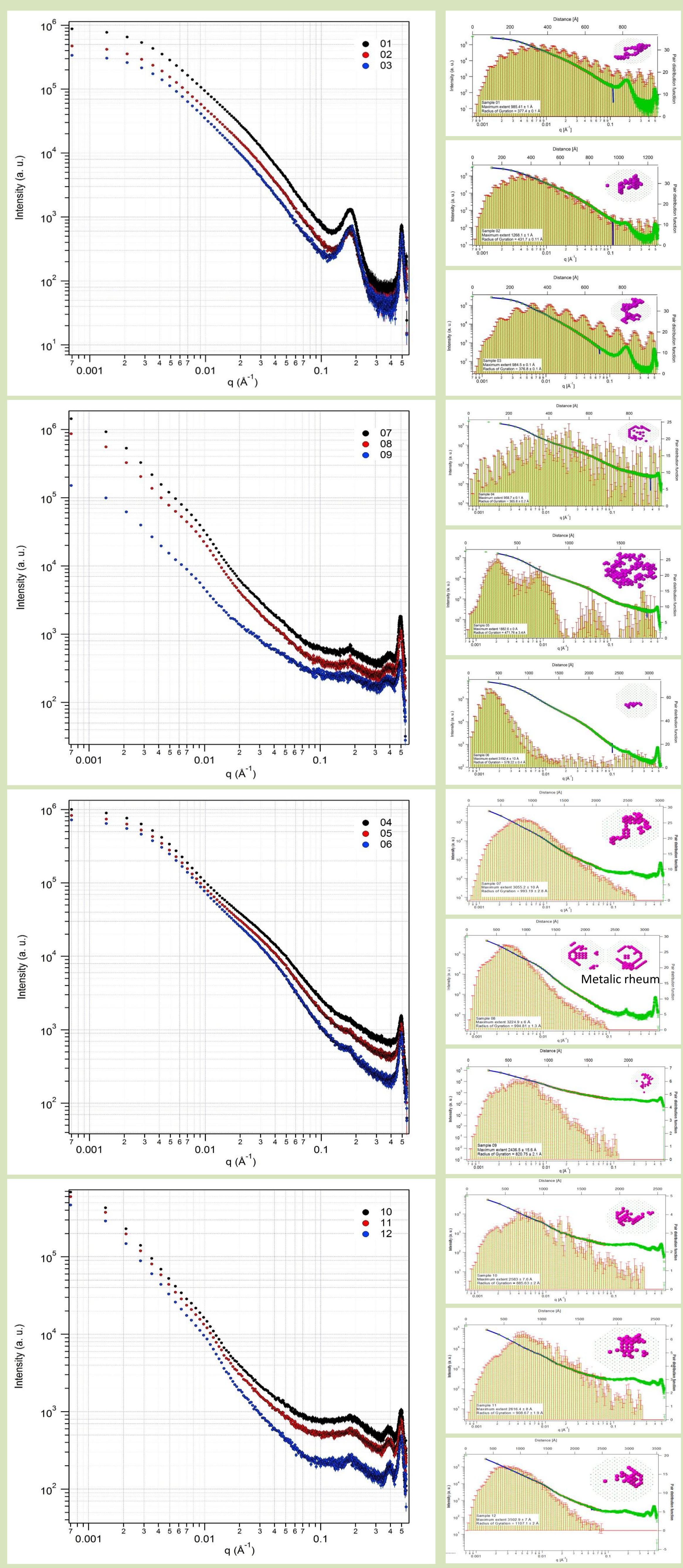


Figure 3. *SAXS results and calculated pair distance distribution functions. PDDFs are given with the DAMMIN outputs as well*

2.4 Experiments

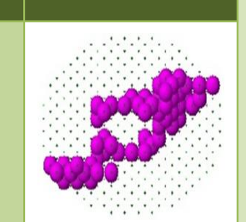
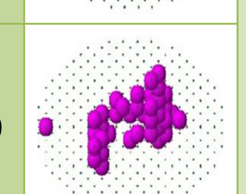
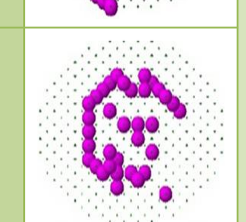
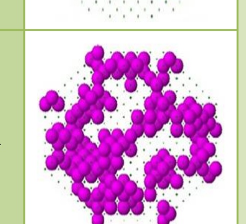
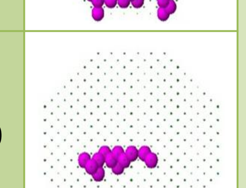
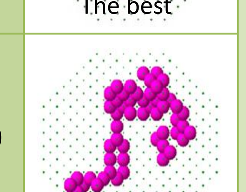
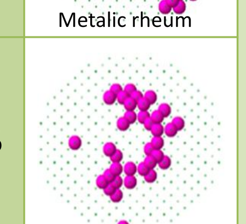
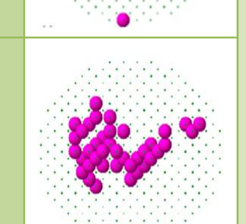
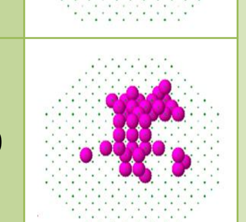
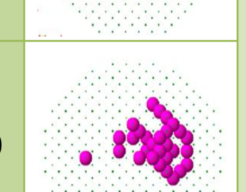
	Sample No.	Radius of Gyration Rg (Å)	Maximum Extend (Å)	Model
Group -1 : Final shape cylindiric, annealing temperature 840º C, production angle 0º, 45º and 90º respectively.	Sample-01	377,4 ± 0,1	985,4 ± 1,0	
	Sample-02	431,7 ± 0,1	1268,1 ± 1,0	 The best
	Sample-03	376,8 ± 0,1	984,5 ± 0,1	
Group -2 : Final shape cylindiric, annealing temperature 0º C, 840º C and 940º C respectively.	Sample-04	365,8 ± 0,2	958,7 ± 0,1	
	Sample-05	471,8 ± 3,4	1882,6 ± 0,1	
	Sample-06	578,2 ± 0,4	3192,4 ± 1,0	 The best
Group -3 : Final shape rectangular prism, annealing temperature 840º C, production angle 0º, 45º and 90º respectively.	Sample-07	993,2 ± 2,8	3055,2 ± 1,0	 The best
	Sample-08	994,8 ± 1,3	3224,9 ± 0,6	 Metalic rheum
	Sample-09	820,7 ± 2,1	2436,5 ± 1,5	
Group -4 : Final shape cylindiric, annealing temperature 0º C, 840º C and 940º C respectively.	Sample-10	885,6 ± 2,0	2583 ± 7,6	
	Sample-11	908,7 ± 1,9	2616,4 ± 8,0	
	Sample-12	1107,1 ± 2,0	3502,9 ± 7,0	 The best

Table 1: DAMMIN outputs.

3. Conclusion

The effects of the production angle on the nano structured implants may be obtained by SAXS analyses. Nano-scale the best prepared samples were indicated Table 1. More uniform PDDs are also evidences of good preparation. Big nano layers are not wanted because of preventing uniform distributions and powerful mechanic properties (depend different orientations of the implants). Beside of internal structure of the samples, surface morphologies are also important. This information will be also recorded with the help of GISAXS measurements at NSRRC-Taiwan. Finally annealing cause the change in nano-structural content of the samples but we need to study the phase transitions of samples by using smaller range of temperature and production angle. Studies about the phase transitions of Ti-6Al-4V samples suggest that 1040º C is critical about α transition [6]. For next generation of samples, production angles such as 0º, 20º, 45º, 70º, 90º and annealing temperatures such as 0º C, 840º C and 1040º C must be further examined. Mechanical properties and XRD experiments are also ongoing to reach collaborative results.

References

[1] Yadroitsev, I. & Smurov, I. Surface morphology in selective laser melting of metal powders. Phys. Procedia 12, 264–270 (2011).
[2] WU, S. et al. Surface nano-architectures and their effects on the mechanical properties and corrosion behavior of Ti-based orthopedic implants. 13–26 (2012).
[3] Necula, B. S., Apachitehi, I., Fratila-Apachitehi, L. E., Van Langelaan, E. J. & Duszcyyk, J. Titanium bone implants with superimposed micro/nano-scale porosity and antibacterial capability. 310–314 (2013).
[4] L. Löber, F. P. Schimansky, U. Kühn, F. Pyczak, and J. Eckert, "Selective laser melting of a beta-solidifying TNMB1 titanium aluminide alloy," J. Mater. Process. Technol., Apr. 2014.
[5] S. WU, X. LIU, K. W. K. YEUNG, H. GUO, P. LI, T. HU, C. Y. CHUNG, and P. K. CHU, "Surface nano-architectures and their effects on the mechanical properties and corrosion behavior of Ti-based orthopedic implants." Surface & Coatings Technology journal, pp. 13–26, 2012.
[6] B. Vrancken, L. Thijs, J.-P. Kruth, and J. Van Humbeeck, "Heat treatment of Ti6Al4V produced by Selective Laser Melting: Microstructure and mechanical properties," J. Alloys Compd., vol. 541, pp. 177–185, Nov. 2012.