SEMESTER REPORT 1

Arfaoui Mohamed (med.arfaoui66@gmail.com)
Supervisor: Prof. György Radnóczi
Co-supervisor: Dr. Viktoria Kovács-Kis,

Ph.D thesis title: Structural characterization of multicomponent alloys and compounds in bulk and thin film form
Place of research: Centre for Energy Research, Thin Film physics Laboratry

A/ Research plan

In the beginning of the semester Prof. Gyor and Dr. Victoria had prepared me to look around the institute (administration stuff and security rules), and to make a general view in the laboratory of magnetron sputtering system and transmission electron microscopy TEM with some documents which describe the system operation, its component rules and calibration of the TEM system (with Victoria), as well as the fundamental structure formation of polycrystalline thin films during deposition and the structure zone models (With Dr György)

After that I got aquainted by Mrs Victoria, with all her research activities are focused in biomaterial and silicate glass corrosion. In order to understand this phenomena I was present to observe the process of the aqueous corrosion experiment with silicate glass samples, made by Victoria, then we examined the structural and chemical interface between the glass and the altered zone using energy filtered transmission electron spectroscopy and energy electron loss spectroscopy. In that period Dr. Victoria, Prof. György and me, we have discussed that recently 45S5 bioglasses in which its compositions are based on the SiO2–Na2O–CaO–P2O5 , have been successfully used as bone materials in dental and orthopedic surgery since when these bioactive glasses were soaked in simulated body fluid which generates a nanocrystalline hydroxy apatite layer (CHA) or hydroxidel apatite layer HA similar to that found in the mineral phase of bone however it has poor mechanical strength , we proposed to focus the research on bioactive glasse-materials for bio-medical application, thereby the question is how to improve its mechanical and bioactivity properties ? In this order the first experiment is tend to identify the HCA layer formation on bioactive glasses with compostion Sio2(45)CaO(25)NA2O(30-x)P2O5(x), x=0,1,3,5 glasse to compare it with different phosphorus pentoxide content rate after immersing in the SBF solution at 37° at different time periods,between 3 hours and 21 days, or maybe it can be amorphous layer which contains the same elements as

in apatite. The SBF solution was prepared according the formula described by KUkubo et al., It can form an amorphous layer which contains the same chemical elements as in apatite. The surface morphologies and elemental composition of the surface layer was examined using Scanning electron microscopy (SEM) with EDS. The experiment was described in the second part of the report.

A second experiment has been done in the beginning of January in which 4 Al films were deposited on Si/SiO2, NaCl(100)and NaCl(100)/collodium substrates under variable Argon flow in a gas mixture of Nitrogen and argon 50%-50% to 50%-70 % by RF magnetron sputtering system. We started by changing the Fe target by the Al target then we put it in the vacuum system. The sputtering system was pumped down to 3 using turbo molecular pumping. The experiment was done and described below.

1/ The in-vitro bioactivity tests of multicomponent oxide glasses in SBF

A -**Material and method**

composition	nominal composition in mole%				
	SiO2	CaO	Na2O	P2O5	sum
S45P0	45	25	30	0	100
S45P1	45	25	29	1	100
S45P3	45	25	27	3	100
S45P5	45	25	25	5	100

A/ Preparation of the SBF solution.

B/ Indicate the interesting area of the sample (as frozen surface) and putting the appropriate volume of SBF into a plastic bottle, then into incubator at 37c° and P(Co2)=0.05atm 5%

C/ Keep the samples into SBF at given condition for different;

3h / 21 days /21 days/ 21 days

D/ Take out each sample and wash it with milli Q water E /

Air_drying the sample without any heating.

Note: the SBF should be used within 30 days after preparation

I participated in the following experiments: S45P0/3h, S45P1/21d, S45P3/21d, S45P5/21d

Figure 1 S45P0-3h

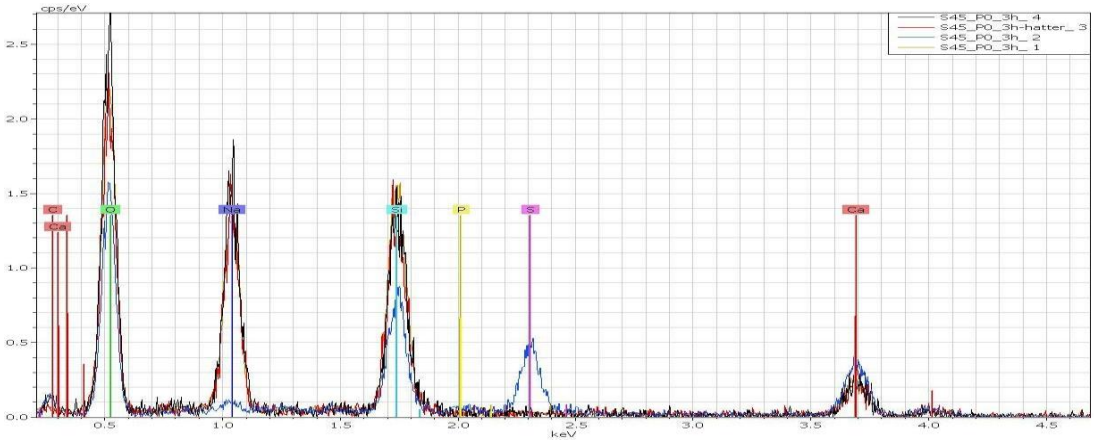


Figure 2 S45P1-21d

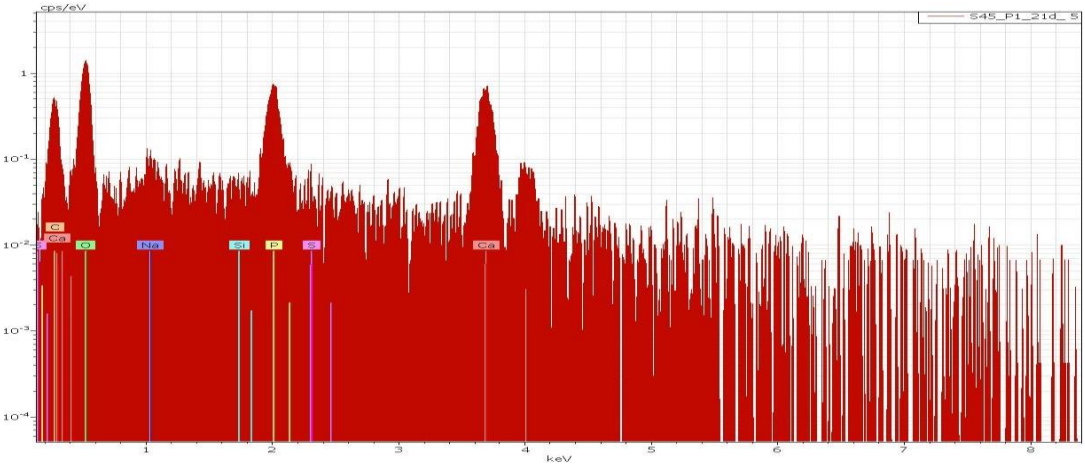


Figure 3 S45P3-21d

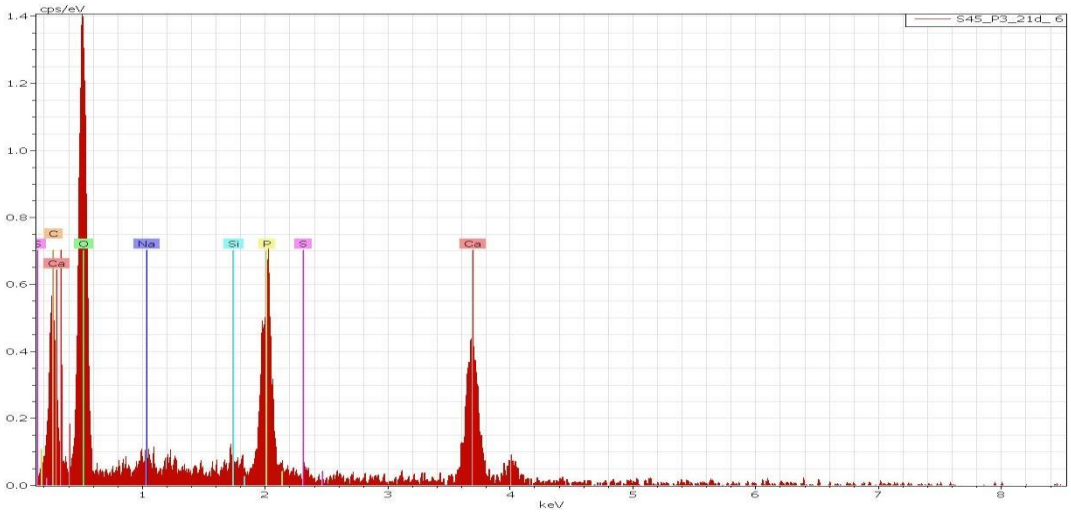


Figure 4 S45P5-21d

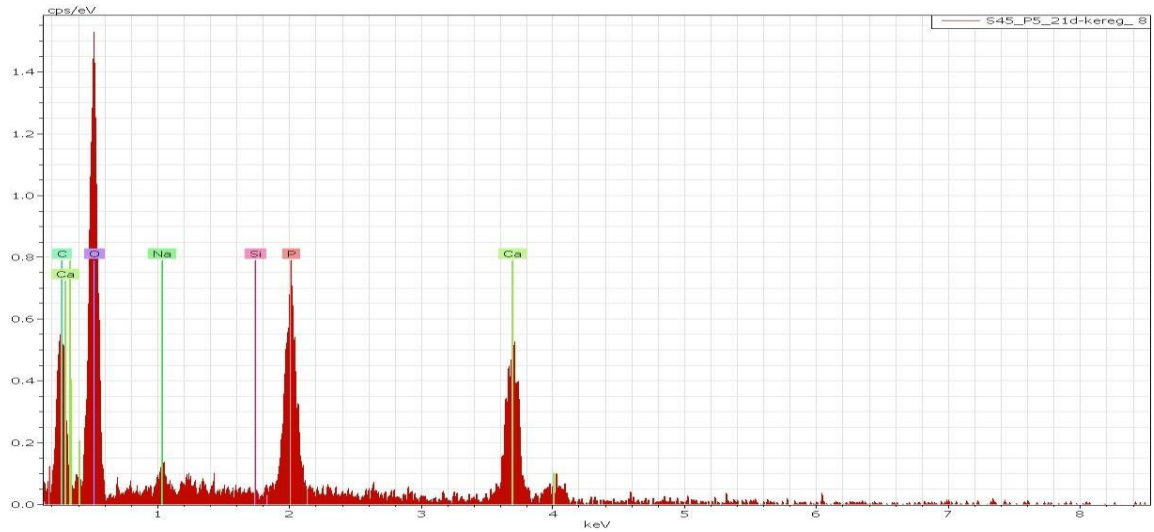
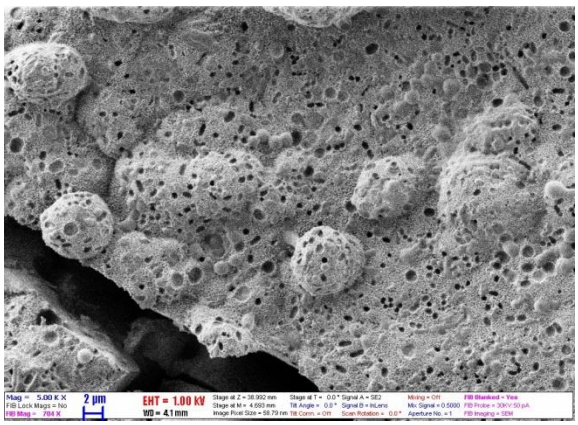
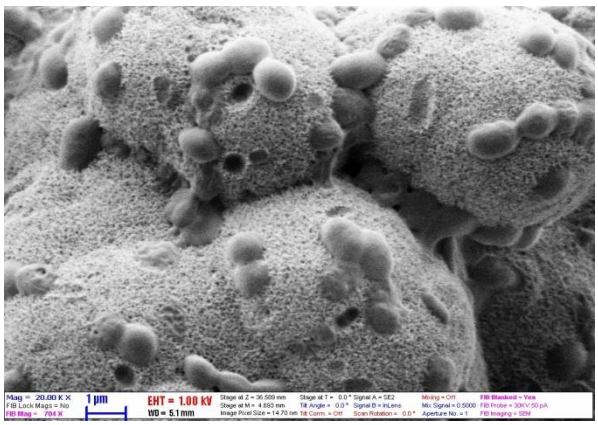
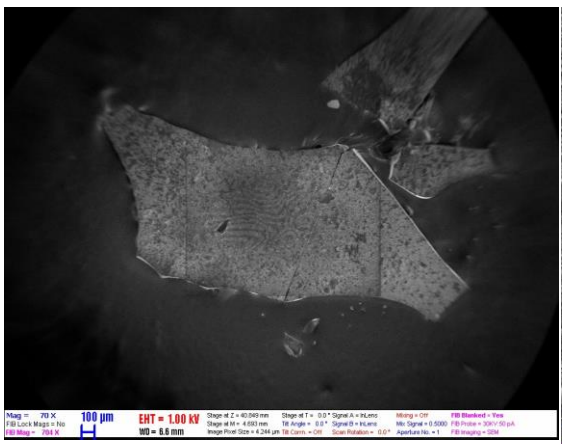


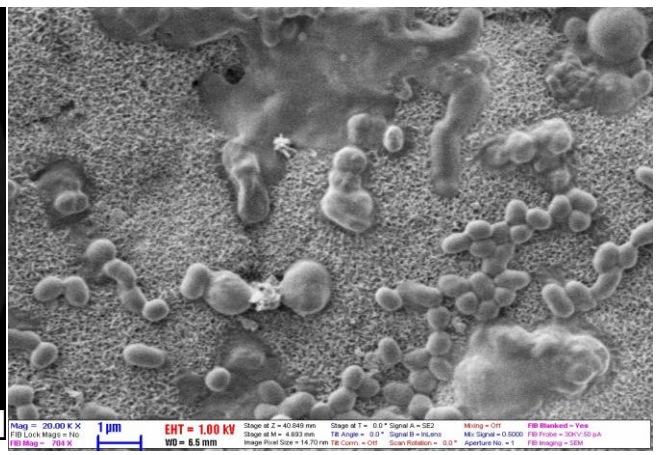
Fig. 5 – SEM micrographs of bioglass samples (A S45P1 21d) (B S45P3 21d) (C ,D S45P5 21d)

A **B**









C **D**

B- SEM and EDS analysis of glass samples

EDS spectra of the samples surface free of phosphorus after immersing in simulated body fluid solution for 3 hours shows no strange element and phase as impurities were found, minor amount of carbone coming from sputtering as well as sulfur and phosphorus indicate ion exchange between SBF and glass. EDS analysis (Fig 1/3/5) shows an increase of phosphorus and calcium rate while there is a decrease of Sodium and silicon rate on the layer with respect to the initial glass composition (Fig 4). The SEM images both of S45p3 and S45P5 were covered with irregular shape of HA particles have been grown into several agglomerates consisting of needle-shaped and boucle-shaped of HA layer (Fig5 A/B/D). This indicates the formation of HA on the surface of the glasse sample after soaking in SBF for 21 days and supports the bioactivity. In case of S45P5 sample there is a significant difference between the carbon content of the bioactive layer and the underlying glass which implies carbonate content in the HA layer. SEM images of P1 show many holes with different size on the bioactive glasses surface .These holes might formed because of the simultaneous formation and degradation of the bioactive layer during the beginning of the soaking period, as it can be seen these holes mostly disappear with increasing phosphate content indicating that high phosphate content favoures layer formation HCA crystals with very small size distributed homogeneously and randomly on the surface and cover holes.

2/ Structural properties of Aluminum Thin Films Deposited by Magnetron Sputtering

Deposition parameters for growth of Al thin film

	Al1	Al2	Al3	Al4	Al5
Substrate	NaCl+Si/SiO2	NaCl+Si/SiO2	NaCl+Si/SiO2	NaCl+Si/SiO2	NaCl/Collodium/Si/SiO2/Colloduim
Total gas (Ar + N2) pressure mbar	**P(Ar): 2.x** 10^{-3}	**P(Ar) 1.x**10^{-3} + **P(N) 1.5**. 10^{-3}	**P(Ar) 1.x**10^{-3} + **P(N) 1.5**. 10^{-3}	**P(Ar) 1.x**10^{-3} + **P(N) 1.5**. 10^{-3}	2.11.10^{-3}
RF power	**140W**	**140w**	**140w**	**140w**	**140W**
Sputtering pressure	**3**10^{-7}	**3.** 10^{-7}	**3.** 10^{-7}	1.6.10^{-7}	1.08 10^{-7}
Deposition time	**20s**	**25s**	**10mn**	**10mn**	**20s**
Structural properties	**Texture d Al**	**Too thin and we could not take it out**	**charging a lot and randomly oriented nanocrystalline**		**Randomly oriented nanocrystalline Al**

B/ Lectures and exams

Diffraction methods in material science: (completed) Dr. Gubicza Jenő

In this lecture we started to learn basic concept of cristallographie and interaction between x-ray photons and material then we went deeply into the size broadening of x-ray line profiles and strain broadenning of x-ray diffraction peak. it was full of theory.

Lattice defects: (completed) Dr. Gubicza Je

This subject contains: Point defects, diffusion, Dislocations and plastic deformation ,Mechanical properties, Stacking and twin faults in face-centered and body-centered cubic crystals,Twinning in hexagonal crystals,Recovery and recrystallization. I

Amorphous alloys: (incompleted) Dr. Révész Ádám

Because of the time and charge of studies I could not complete it

Exams

The exams regarding both of the courses (diffraction methods in materials science and lattice defects) will be taken on 18 and 21 of january. For the amorphous alloys courses not yet .

In this semester, unfortunately I got robbed about one month ago and I lost everything (documents, money and phone). That's made this a very hard semester. Moreover my accommodation is too far from the laboratory, I spend one hour and half to get there. I am asking with pleasure if maybe there is a possibility to change the dormitory