4. semester report

Péter Nagy (nagyp@student.elte.hu)

PhD program: Materials science and solid state physics

Supervisor: Jenő Gubicza

Thesis title: Correlation between processing, microstructure, and mechanical properties of novel multicomponent thin films

Introduction:

In the last decades, several novel multicomponent materials comprising 3-5 different chemical elements were developed. For instance, medium or high-entropy alloys (MEAs or HEAs) contain at least four components, usually with equal fractions. These structures are stabilized by the large configuration entropy. Due to the severe lattice distortion and the sluggish diffusion, HEA materials exhibit high strength, good ductility as well as excellent corrosion resistance and thermal stability. Therefore, HEAs are considered as advanced structural materials with outstanding mechanical properties. HEAs are intensively studied materials due to their impressive mechanical properties, such as very high strength even at high temperatures. HEAs with many different compositions have already been successfully processed in bulk form. The synthesis of HEA materials exploits many different methods such as melt spinning, electromagnetic stirring, vacuum arc melting, or mechanical alloying. However, there is a demand for the production of these materials in the form of thin films as they can be used as hard coatings in many practical applications. During my MSc studies, I worked on the production of MEA thin films and participated in an elaboration of a novel physical vapor deposition method. In my PhD work, I demonstrated that MEA thin films can be processed using a multiple beam sputtering system in PVD, which does not require preliminary manufacturing of MEA targets, but rather uses commercially pure metal targets. This study also demonstrated the capability of this new multiple beam sputtering technique for the production of compositional gradient samples with a wide range of elemental concentrations, enabling combinatorial analysis of multiple elements high-entropy alloy. The effect of the chemical composition on the structure and properties of MEA/HEA films can be studied on combinatorial samples. We used synchrotron X-ray diffraction to create a diffraction map for one of these gradient samples, thereby we can examine the changes of the microstructure as a function of the chemical composition.

Research work in the previous three semesters:

CoCrFeNi samples were produced by multiple beam sputtering physical vapor deposition. The mechanical properties were characterized by nanoindentation, and the microstructural properties were investigated by synchrotron X-ray diffraction. The chemical composition of the thin film was investigated by energy-dispersive X-ray spectroscopy (EDS) and X-ray fluorescence spectroscopy (XRF).

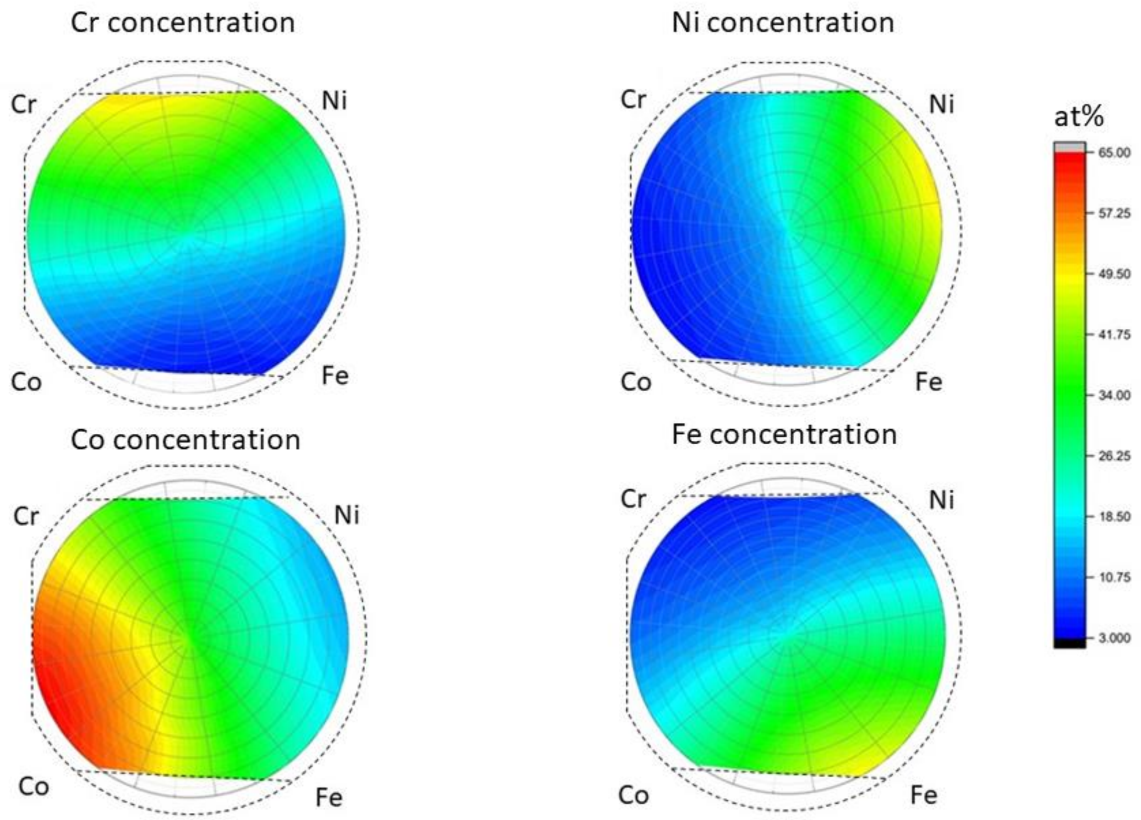

Figure 1: Element maps of a CoCrFeNi thin layer as obtained by XRF.

The samples produced by multiple beam sputtering PVD method had nanocrystalline microstructure with crystalline size between 10-20 nm. Very high dislocation density (60–280 $\times 10^{14}$ m^{-2}) and twin fault probability (0.9–4.6%) were observed for all compositions of the combinatorial layer. For most of the thin film, a single-phase fcc structure was formed as shown in Fig. 2. Close to the periphery of the disk sample between Fe-Co and Co-Cr sources, the main phases were bcc and hcp, respectively. It was revealed that the highest hardness (~11.8 GPa) was achieved for the composition 42% Co–45% Cr–5% Fe–8% Ni (at.%). In this point, the major phase was hcp which has a lower number of easy-slip systems compared to fcc or bcc structures, resulting in a higher hardness. In all fcc points, a strong 111 fiber texture was observed. For the hcp phase, both 100 and 101 texture components co-exist. Between Fe and Co PVD sources, where a single-phase bcc structure was identified, the texture was characterized by a stronger 111 and a weaker 110 components.

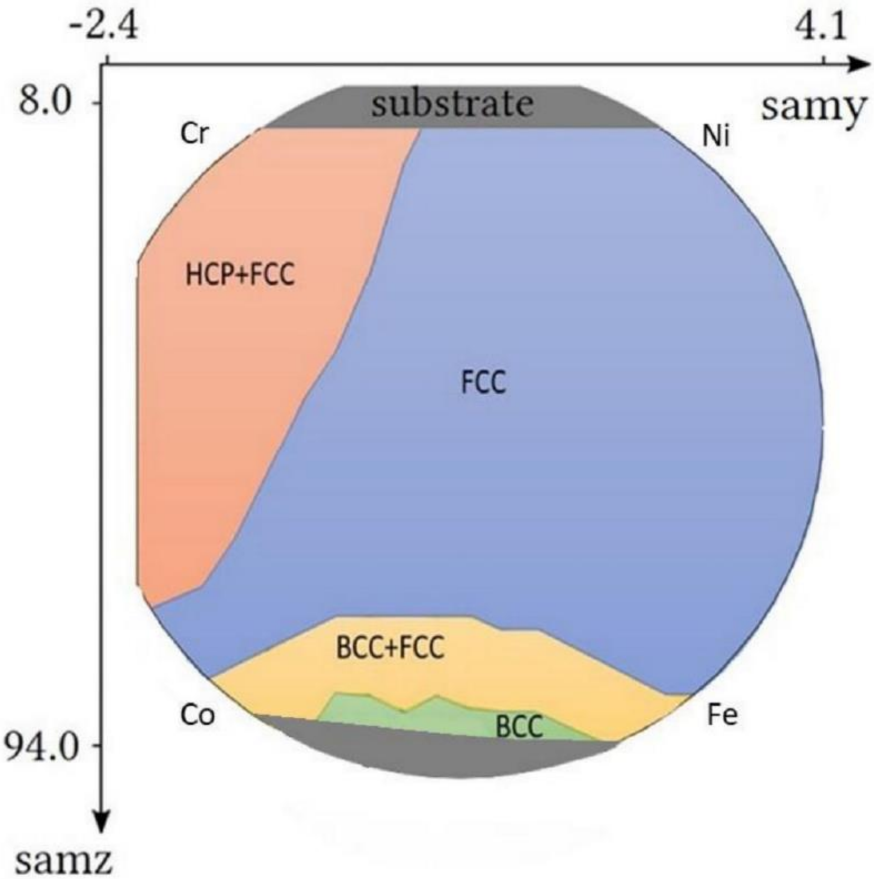

Figure 2: Phase map of a CoCrFeNi thin film as determined by synchrotron XRD.

We also started to produce MEA thin layers via electrolytic deposition in the Wigner institute. Due to the lack of vast literature on the electrochemical processing of MEA/HEA thin films, the effort to create such layers and understand the procedure is motivated. Furthermore, the samples will help to understand the processing-specific mechanical and microstructure properties of the PVD samples.

Also, I participated in many other projects during my first three semesters, such as:

- the investigation of the microstructure of severely deformed ZK60 Mg alloys by X-ray diffraction and SEM
- the investigation of the microstructure of severely deformed AA1050 Al alloys by X-ray diffraction and SEM
- the investigation of the microstructure of severely deformed AA5083 Al alloys by X-ray diffraction and SEM

Research work in the current semester:

- We managed to produce electrolytic MEA thin layers which are free of cracks and other surface defects, therefore we can proceed with the mechanical and microstructural investigation of the samples.
- In this semester, I visited the Deutsches Elektronen-Synchrotron (DESY) two times to carry out in-situ X-ray diffraction measurement during annealing and uniaxial tensile

test on Mg-LPSO (Long-Period Stacking/Order) samples in the frame of our MagMAX V4-Japan project.

Publications related to my PhD topic:

- Nagy, P.; Rohbeck, N.; Hegedűs, Z.; Michler, J.; Pethö, L.; Lábár, J.L.; Gubicza, J., Microstructure, Hardness, and Elastic Modulus of a Multibeam-Sputtered Nanocrystalline Co-Cr-Fe-Ni Compositional Complex Alloy Film. *Materials* **2021**, *14*, doi:10.3390/ma14123357. (ranking: Q1, IF: 3.6)
- Nagy, P.; Rohbeck, N.; Widmer, R.N.; Hegedűs, Z.; Michler, J.; Pethö, L.; Lábár, J.L.; Gubicza, J., Combinatorial Study of Phase Composition, Microstructure and Mechanical Behavior of Co-Cr-Fe-Ni Nanocrystalline Film Processed by Multiple-Beam-Sputtering Physical Vapor Deposition. *Materials* **2022**, *15*, 2319, doi:10.3390/ma15062319. (ranking: Q1, IF: 3.6)

Publications not related to my PhD topic:

- Sidor, J.J.; Chakravarty, P.; Bátorfi, J.G.; Nagy, P.; Xie, Q.; Gubicza, J.; Assessment of Dislocation Density by Various Techniques in Cold Rolled 1050 Aluminum Alloy. *Metals* **2021,** *11*, 1571, doi:10.3390/met11101571. (ranking: Q2, IF: 2.4)
- Sabbaghian, M.; Fakhar, N.; Nagy, P.; Fekete, K.; Gubicza, J. Investigation of shear and tensile mechanical properties of ZK60 Mg alloy sheet processed by rolling and sheet extrusion. *Mater. Sci. Eng. A* **2021**, *828*, 142098, doi:10.1016/J.MSEA.2021.142098. (ranking: D1, IF: 5.2)
- Fakhar, N.; Sabbaghian, M.; Nagy, P.; Fekete, K.; Gubicza, J. Superior low-temperature superplasticity in fine-grained ZK60 Mg alloy sheet produced by a combination of repeated upsetting process and sheet extrusion. *Mater. Sci. Eng. A* **2021**, *819*, 141444, doi:10.1016/J.MSEA.2021.141444. (ranking: D1, IF: 5.2)

Paper under review

- N. Fakhar, E. Khademi, M. Sabbaghian, A. Momeni, F. Fereshteh-Saniee, P. Nagy, Á. Szabó, J. Gubicza, Microstructure and hot shear deformation behavior of a fine-grained AA5083 aluminum alloy processed by DECLE, Materials Chemistry and Physics (2022) submitted. (ranking: Q2, IF: 4.094)

Conferences:

- OATK2021-XIII Országos Anyagtudományi Konferencia – Oral lecture „Nagyentrópiás ötvözet kombinatorikus vékonyréteg előállítása és karakterizálása", 2021.10.10. Magyarország, Balatonkenese

- 2nd World Congress on High Entropy Alloys (HEA 2021) Oral lecture „Preparation and characterization of a HEA thin film combinatorial sample." 2021.12.5. USA, North Carolina, Charlotte

Studies in the current semester:

subject code	subject name	course type	number of classes	number of credits	Lecturers	Grades
FIZ/1/022E	Solid state theory I.	Lecture	2	6	Groma István	Excellent (5)

Teaching:

1. semester:

subject code	subject name	course type	number of classes
applphysf17lm	Methods of Applied Physics Laboratory	Laboratory	4

2. semester:

subject code	subject name	course type	number of classes
fizlab3f19la	Modern Physics Laboratory	Laboratory	4

3. semester:

subject code	subject name	course type	number of classes
applphysf17lm	Methods of Applied Physics Laboratory	Laboratory	4

4. semester:

subject code	subject name	course type	number of classes
fizlab4f19la	Modern Analytical Methods	Laboratory	4