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http://lcpmr.cnrs.fr/content/interfaces-mutimatériaux-sources-et-optique-x

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Report on PhD manuscript "Study of influence of a barrier layer material and annealing temperature on the formation of interlayer regions in short-period multilayer mirrors based on beryllium" by SAKHONENKOV Sergei Sergeevich

The work deals with the characterization of innovative W/Be multilayers designed as optical components for the soft x-ray range. These multilayers should have small periods, of a few nanometers, so the question arises about the quality of the interfaces. Indeed, too large roughness or too strong interdiffusion can lead to a loss of the optical contrast between the different layers and then to a decrease of the optical performances. So, in his manuscript, Mr. SAKHONENKOV presents a comprehensive knowledge-based approach of the interfaces of W/Be and Cr/Be stacks, relying mainly on characterizations performed by x-ray photoelectron spectroscope, so that the x-ray optical properties can be anticipated from the interface characterization.

In Chapter 1 the candidate presents some consideration regarding x-ray optics and particularly about periodic multilayer designed as optical components for the x-ray spectral range. The fabrication of these devices is also described and emphasize is put on magnetron sputtering, the technique used to prepare the studied samples. Details are given on the physical phenomena taking place during the deposition process, which will be useful in the following chapters to interpret the results. Then, the main analysis technique used in this work, x-ray photoelectron spectroscopy, is introduced. Its principles are detailed and particularly the way the binding energies of the core level can shift is discussed. But XPS is not a technique limited to the analysis of core levels. I think that a comment justifying why only core levels were used and why valence bands were not considered at all would have been welcome.

The following chapter gives the technical details on the apparatus used to prepare the sample and analyze them. It is impressive to see that the candidate had access to many XPS apparatus, either in laboratories or on synchrotron beamlines. Then x-ray reflectometers are presented. Here again, I can see that laboratory and synchrotron apparatus have been used. I have been impressed by the long list of samples, single layers, bilayers, trilayers or periodic multilayers, given with the details of their deposition conditions, and needed owing to the methodology set up to understand all the interfaces of the W/Be system.

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With Chapter 3 begins the presentation of the results and their interpretation. Thus, the interfacial phenomena taking place in the W/Be and Cr/Be structures are discussed, going from the reference samples to the more complicated ones, the bilayers, for which the order of the layers can be inverted in order to get access at the A-on-B interface or B-on-A interface. The complexity keeps going until the analysis of periodic multilayers. This first part of the chapter relies on the core level XPS spectra obtained from the different samples and their decomposition into different chemical components. My main remark comes from the fact that I see in some tables that the width of a core level can change with the chemical state of the considered element. This is not expected from the explanation of the physical principles of the XPS technique given in Chapter 1 and would have deserved discussion.

Then a detailed description of the possible compounds able to form at the interfaces is given. It is based on a bibliographic study involving mainly thermodynamic considerations. It seems that the data do not exist for the W-Be and Cr-Be systems and thus the candidate relies on a comparison with the Au-Al system. I think that some comments explaining why the Au-Al results can be transferred to W-Be and Cr-Be would have been welcome. Following, some simulations of how the ions propagate into materials are explained and given in order to understand how the elements coming from the sputtering targets impinge the surface of the substrate when the deposition process occurs. This enables to understand the difference between W-Be and Cr-Be systems, but also within a system why interfaces can be asymmetric.

After a short discussion about the oxidation of the samples, the case of periodic multilayers is addressed. Here HAXPES is used to get deeper information. I have found some part of this discussion to read because some formed compounds are named BeW and other WBe depending on the stoichiometry. Changing the order of the elements from one compound to another was really troublesome for me and made the reading of this part of the manuscript difficult.

Following the study of the "simple" systems, in Chapter 4 the candidate discusses the insertion of B_4C and Si thin barrier layers to improve the stability of the considered stacks. At the beginning of chapter are given numerous conditions that these barrier layers have to fulfill but none are related to the x-ray properties. It is deduced here that the barrier layers lead to more symmetrical interfaces and to more contrasted interfaces from the x-ray optics point of view. After a short discussion of the possible phenomena taking place after insertion of the barrier layers, the annealing of the samples is discussed. The XPS analysis clearly demonstrates, with respect to the original systems, that additional interfacial mixing does not occur upon annealing when B_4C is used as a barrier layer, whereas the insertion of silicon as barrier layer decreases the formation of interfacial compounds. In all cases, the oxidation of the surface of the samples is observed.

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Chapter 5 deals with the simulation and measurements of the optical properties in the soft x-ray range of the considered W/Be stacks, with or without barrier layers, with or without annealing. The Cr/Be system is not considered here. I found that it is a pity that simulations rely mainly results obtained from electron microscopy rather than photoelectron spectroscopy. As expected the existence of the interlayers decreases the reflectance of the W/Be stacks. The insertion of the B_4C barrier layer is beneficial from this point of view. The reflectivity decrease as a function of the annealing is smaller when barrier layers are introduced in the stacks, demonstrating the improvement of the thermal stability in this case.

The manuscript ends up by a conclusion which is a mere repetition of the conclusions of the previous chapters and by an impressive list of more two hundred references.

In spite of the few remarks that in no way question the quality of the work done, I would like to stress the strategy of the candidate who to better analyse the W/Be system. He first studied simpler systems with only one, two or three periods and finally characterize periodic multilayers. This led to a considerable number of samples and to numerous XPS analysis, performed on a large number of apparatus, which Mr. SAKHONENKOV performed with rigour and method, and a convincingly presented in a clear way. Owing to the quantity, quality and scientific interest of the presented work, I think that Mr. SAKHONENKOV has written a manuscript worth of a PhD degree and that he deserves the obtention of such a degree.

Dr. Philippe JONNARD