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Prof. dr hab. Krystyna Jabłońska
Instytut Fizyki
Polskiej Akademii Nauk

Recenzja poprawionej rozprawy doktorskiej mgr Petera Ershov'a pt.

„Wysoka rozdzielczość dyfraktometria rentgenowska i reflektometria półprzewodnikowej struktury mikro- i nano- oparciu o rentgenowskich refrakcji optyki"

przygotowanej pod kierunkiem dr Anatoly'a Snigirev'a i dr hab. Anny Perelomovej

tytuł angielski

„High resolution X-ray diffractometry and reflectometry of semiconductor nano- and micro- structures based on X-ray refractive optics"

Rozprawa została uzupełniona i poprawiona z uwzględnieniem większości uwag zawartych w przekazanej przeze mnie w październiku 2015 recenzji. Wraz z rozprawą w postaci manuskryptu otrzymałam spis dokonanych poprawek oraz streszczenie w języku polskim. Streszczenie w języku polskim wymagane jest w przypadku rozpraw, które zostały przygotowane w języku innym niż polski. Rada Naukowa prowadząca przewód doktorski wyraziła na to zgodę. Streszczenie w języku polskim zostało przygotowane za pomocą internetowego tłumacza, bez konsultacji z kimkolwiek kto zna język polski i nie nadaje się do upublicznienia, podobnie jak podany tytuł rozprawy w języku polskim. Rozumiem, ze zarówno autor rozprawy jak i promotorzy nie znają języka polskiego, mam jednak nadzieję, że zostanie to poprawione w oficjalnych dokumentach.

Po zapoznaniu się z uzupełnioną i poprawioną rozprawą stwierdzam, że w obecnym kształcie rozprawa spełnia w sposób zadawalający wymogi określone w ustawie o stopniach naukowych:
„Rozprawa doktorska, przygotowywana pod opieką promotora albo pod opieką promotora i promotora pomocniczego, o którym mowa w art. 20 ust. 7, ***powinna stanowić oryginalne rozwiązanie problemu naukowego*** *lub oryginalne dokonanie artystyczne* ***oraz wykazywać ogólną wiedzę teoretyczną kandydata w danej dyscyplinie naukowej*** *lub artystycznej oraz* ***umiejętność samodzielnego prowadzenia pracy naukowej*** *lub artystycznej.*

W dalszej części mojej oceny postaram się uzasadnić powyższe stwierdzenie. Widząc kłopoty autora z językiem polskim sformułuję uzasadnienie w języku angielskim.

The PhD dissertation is devoted to exploitation of the refractive lenses which provide one and two dimension Fourier transform, in high resolution X-ray diffraction (HRXRD) and reflectometry (XRR). The main aim of the thesis defined by author is:
"To modernize the existing HRXRD and XRR methods by increasing the angular, spatial and time resolution for present-day synchrotron sources."
Other objectives are the following:

1. *To analyze conventional X-ray high-resolution methods of diffractometry and reflectometry.*
2. *To design and implement an alternative X-ray optics scheme of HRXRD and XRR based on refractive X-ray lenses.*
3. *To perform experiments and process data in order to generate the results demonstrating the advantages of new methods.*

   Two concepts should be studied experimentally in the thesis:

   I. *X-ray Compound Refractive Lenses Fourier Transform (CRL FT) for HRXRD tasks.*
   II. *Hard X-ray Reflecto-Interferometer (HXRI) for X-ray Reflectometry tasks.*

The aims of the thesis are therefore well defined, are very ambitious and are related to the improving very popular techniques extensively exploited in nanotechnology and material science. The idea of refracting lenses is known since 1995 and the refractive lenses become used in the X-ray optics after successful demonstration of excellent focusing and imaging properties in the first publication of Anatoly and Irina Snigirev at al. in 1996. Moreover, Dr Anatoly Snigirev is the supervisor of the thesis.
The use of X-ray refractive lenses has rapidly expanded and consequently they are now widely exploited on synchrotron beamlines forming easy to implement and operate in-line optics. The lenses used by the author in presented experiments, the compound refractive lenses (CRL), have a number of advantages as compared to classical optics. They are easy to align, exhibit stability and are relatively insensitive to misorientations and mechanical vibrations. It is listed in the thesis and is well recognized in the X-ray community. The transfocators, used by author in experimental set-up and described in the updated thesis, offer focal length tenability that drastically extends the applicability of refractive optics. The CRL can be adapted to very high X-ray energies by modifying composition and number of lenses, and furthermore, refractive optics can be easily inserted and removed from the beam to allow fast switching the beam size from the micrometer to nanometer scale. I was expecting that all these properties will be comprehensively discussed in the thesis showing the theoretical knowledge of author in this particular discipline and implemented to the X-ray diffraction and reflectivity measurements and the results compare to the classical set-up.
The thesis is presented in 86 pages (first version 51) and beside Introduction and Conclusions and Outlook, is divided into four chapters. The author refers to 64 publications. The first two chapters demonstrate the general knowledge of the author regarding the X-ray sources, optics, X-ray diffractometry and reflectometry. The overview of the X-ray sources is provided including the free electron lasers. The modern X-ray optics is also well presented together with general description of the idea of compound refractive lenses and transfocator. Due to

the fact that refractive lenses are the core of the dissertation, I think that they should be described more comprehensively. The dependence of their parameters on the material used and energy of radiation is only mentioned but not considered in the relation to studied samples and used configuration. That means that the objective no 2. *"To design and implement an alternative X-ray optics scheme of HRXRD and XRR based on refractive X-ray lenses"* is not very deeply argued. Author in the presented experiments is using the lenses from different materials (Si, Be) with altered numbers of lenses in stack (from 3 to 51) and several radiation energy. The question how this choice was made and how it influences the experiment is not discussed.

The X-ray diffractometry and reflectometry is presented in second chapter. It is related to objective no1.The general principles of these techniques are well described on 10 pages together with introduction of the concept of reciprocal space. Information which usually are gained from these techniques are only listed. Again, the main goal of the thesis is to modernized these two techniques, so I think that author should more deeply consider the advantages and shortcomings of these classical methods. Nevertheless, from the presented description I can confirm that author has general theoretical knowledge about these methods according to requirements for the PhD thesis.

In the Chapter 3 with title "X-ray optical approach for high resolution diffractometry and reflectometry" author considered theory of visible light FT in the case of in-line geometry according to description provided in reference [38] and discussed the changes introduced in the Bragg geometry. The predictions of geometrical optics do not include the effects of diffraction. After consideration of these effects, the possibility to sweep reciprocal space is shown what evidences the potential of these lenses for HRXRD. Nevertheless, no comparison between accuracy which can be achieved in the traditional and proposed approach for studies in reciprocal space is provided. However, sensitivity of the FT technique to deformation is considered on example of binary square-wave phase Si grating and was found to be equal to $\Delta d/d = 10^{-5}$. If we recall that in Chapter 2 for classical method it was 10^{-4} it is one order of magnitude better.

The concept CRL exploitation in the X-ray microscopy is briefly described with the reference to the paper in which the author participated as a specialist in X-ray microscopy, but the results are not discussed. Therefore these results do not belong to the PhD thesis but may evidence that according to requirements for PhD author has ability to perform independently scientific studies. The potential of using the CRL in the reflectometry is indicated. Nevertheless, all of these may take place in the future and the only drawback indicated is high X-ray intensity which can destroy the membrane. I think that should be other reason that despite that CRL are known over 20 years they are still not widely used in X-ray reflectometry and microscopy. The Chapter 4 presents the "Experimental Results", which should be the **original solution of the scientific problem**. The presented experimental results for HRXRD have been published in the pre-review journals [e.g.12 and paper 3 p.80] and the author is the first in the list of authors. Therefore, these papers have to report new, original solution of scientific problems. The HRXRD results are presented for one micro- and one nano- structures, the HXRI results for Si_3N_4 membranes with 3 thicknesses and 100 nm PMMA film.

The experimental set-ups were described with all important details. Measurements were performed at ESRF on beamline ID06 with the parabolic Be CRL with curvature R =50 μm (it is not clear if it is transfocator indicated in Fig.48?) as a "more efficient X-ray optics in the chosen X-ray spectra". It is the only consideration regarding the most important for the thesis part of X-ray optics. Next two Si microstructures, the studied objects, are described and parameters of experimental set-up are listed. The Si lenses with different parameters are used in this case. I suppose, that together with set of lenses described before. The author claims that " by analyze the reciprocal space map we can conclude about different nature of imaging contrast formation" for both studied structures. Next, the minimum oxide strip thickness to resolve is estimated (40 nm). Simulation of the reciprocal space are presented without information what kind of the code were used and what mathematical model of displacement or strain (which author claim was not exactly correct) was used. Finally, I do not see what was the different nature for formation imaging contrasts in these two structures. Only after reading the paper [12] I have got idea about results of performed studies. In summary, interesting experiment but not comprehensively described in the thesis. Moreover, this part was added in the corrected version of thesis.

The second experiment was performed on a Si-Ge nano-heterostructure. In this experiment author is also using the geometry: lenses before object, with transfocator located closer to source (but closer in relation to what?). From Table 3 it is L=39 m and f=1965 cm (19,65m), but in experiment scheme it look different (Fig. 55). The choice of cartridge with two kinds of lenses (I think in this case Be) is again not explained at least the finally used CRL were described. The position of lenses for the minimal source size was found and the period of the Si pillars, which was in agreement with nominal vertical period for the structure was measured. The horizontal period needs to be corrected for stretching. The achieved results confirmed that the proposed technique can be used for measuring the periods in the heterostructures and "dynamical experiments can be performed". Nevertheless, any proposition of dynamical experiment is discussed. FT pattern on the larges area detector are presented for observation of XRD on germanium islands but next it is claimed that the Si pillars are responsible for formed satellites at reciprocal space and the Ge islands are not mentioned. In the description of experiments several this kind of inconsistencies are present. Since the lenses geometry before the object introduces difficulties in interpretation of the reciprocal space, the geometry with the lenses after object was tested. The number of Be lenses was now 50 (instead of 10 and energy of used radiation was 15 keV). Only in this geometry Ge (400) reciprocal point was detected on a large area detector and halo corresponding to the defected Ge crystal structure was indicated. According to main aim of the thesis, which is: *"To modernize the existing HRXRD and XRR methods by increasing the angular, spatial and time resolution for present-day synchrotron sources"*, in conclusions the main parameters of new approach and conventional one are compared. Parameters provided in Table 7 and 8 in my opinion were not well explained. It is not clear for me why the achieved resolution in real space 0.2 mm can allow to analyze microstructural gird with periodicity up to 8 μm or analysis locality 40 μm in HXRI. Finally, the degradation of PMMA was observed in time scale of 0.5 s.

To summarize my opinion, I state that the author performed three kinds of experiments using CRL in HRXRD and HXRI mode. The results for HRXRD have been already published, therefore the original solution of scientific problem was well demonstrated. Very general knowledge of the author in the field of X-ray optics was shown in first three chapters of thesis. The skill to perform independently the scientific work is difficult to evaluate, but from the list of publication (page 80) where author was participated I can state that he was exploiting there the knowledge about the properties of CRL. Therefore, all three requirements for PhD thesis are fulfilled and the author can proceed with the other steps of the PhD program.

All my criticism is related to the way the results have been presented. In many cases without comprehensive discussion or explanation of the Figures and Tables contents. To some extent, it can be attributed to the fact that thesis were written not in author's native language.