# Structure and mechanical stresses in TaSi<sub>2</sub>/Si multilayer

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The structure, construction and mechanical stresses of the  $TaSi_2/Si$  multilayer manufactured by magnetron sputtering on the heated substrates were studied in the initial state and after thermal annealing. Deposition of multilayers is accompanied by formation of compressive mechanical stress. Reduction of mechanical stresses in the multilayers by increasing of the substrate temperature and after thermal annealing was observed. The thickness of the multilayers and the annealing temperature at which ones separated from the substrate were shown. Thermal annealing is accompanied by changes of the multilayer construction and structure of layers. The deposition of depth graded multilayers with layer thickness distribution according to the Fresnel zone plate law has been performed.

Keywords: multilayer, magnetron, zone plate, mechanical stress, crystallization.

В исходном состоянии и после термического отжига исследованы структура, конструкция и механические напряжения многослойного покрытия TaSi<sub>2</sub>/Si, изготовленного магнетронным распылением на подогреваемые подложки. Нанесение покрытия сопровождается формированием сжимающих механических напряжений. Наблюдалось уменьшение механических напряжений в покрытии при возрастании температуры подложки и после термического отжига. Показана толщина покрытия и температура отжига, при которых происходит отделение покрытия от подложки. Термический отжиг сопровождается изменением конструкции покрытия и структуры слоев. Проведено нанесение покрытия с распределением толщины слоев по глубине согласно уравнению Френеля для зонных пластинок.

Структура та механічні напруження у багатошаровому покритті TaSi<sub>2</sub>/Si. О.Ю.Девізенко, І.А.Копилець, В.В.Кондратенко, Ю.П.Першин, І.Ю.Девізенко, Е.М.Зубарєв, Б.А.Савицький.

У початковому стані та після термічного відпалу досліджено структуру, конструкцію і механічні напруження багатошарового покриття TaSi<sub>2</sub>/Si, яке виготовлено магнетронним розпиленням на підкладки, що підігрівали. Нанесення покриття супроводжується формуванням стискувальних механічних напружень. Спостерігалося зменшення механічних напружень у покритті при зростанні температури підкладки та після термічного відпалу. Показано товщину покриття та температуру відпалу, при яких відбувається відокремлення покриття від підкладки. Термічний відпал супроводжується зміненням конструкції покриття та структури шарів. Проведено нанесення покриття з розподілом товщини шарів у глибину відповідно рівнянню Френеля для зонних платівок.

#### 1. Introduction

The linear zone plate (LZP) is an optical element for focusing X-rays, in our case with a photon energy E > 10 keV. The basis of the LZP is a multilayer [1], in which, due to a significant difference in the absorption coefficients, one material transmits X-ray along the layers, and the second material absorbs radiation. In such a multilayer, the thickness of the layers in the direction from the substrate to the top layers corresponds the Fresnel zone plate law [1]. Among the number of different requirements imposed to multilayer for LZP, the necessity to deposit a large number of layers and provide sharp layer interfaces are the part of ones.

As previous studies have shown [2, 3], the  $W_5Si_3/Si$  multilayer has limited possibilities to form LZP. This is due to the appearance of high-level compressive mechanical stresses during the deposition of the multilayer, which restricts the multilayer maximum thickness to about 16 µm. Thicker multilayers exfoliated from the substrates or the substrates cracked. To reduce the mechanical stresses by structural relaxation in Si layers the multilayer was annealed. Due to the phase non-equilibrium of this system, interface mixing during annealing was observed, and that led to a change in the ratio of the layers thickness and the period. To compensate such changes, additional correction was required in the layer thickness ratio at the deposition stage. The next step is to consider the TaSi<sub>2</sub>/Si phase-equilibrium multilayer [4] in which no interface mixing in the initial state and after thermal annealing is expected. To create an effective LZP based on

Table. Parameters of multilayers

the TaSi<sub>2</sub>/Si multilayer, it is required to investigate the features of its growth, including the interface roughness, the presence and value of mechanical stresses, and effect of annealing on the change of multilayer parameters. Silicides of W and Ta have close values of the absorption coefficient of X-ray  $radiation \ with \ a \ wavelength \ of \ about$ 0.1 nm. Therefore, the transition to the TaSi<sub>2</sub>/Si multilayer will not lead to a significant change in the design of the LZP, and its optical properties. Thus, the purpose of this paper is to investigate the possibility and features of the formation of TaSi<sub>2</sub>/Si multilayer with a layer thickness of  $2\overline{5}$  – 33 nm and a total thickness of more than 16 µm for a LZP working with X-ray energy E > 10 keV.

#### 2. Experimental

The multilayers were fabricated by DC magnetron sputtering of  $TaSi_2$  (99.9 %) and Si (99.99 %) targets under Ar pressure of about 0.2 Pa. There were prepared 2 types of multilayers: 1) multilayers (Table, No.1-7) with constant period and equal thicknesses of both layers and 2) depth graded multilayers (Table, No. 8, 9) with layer thickness distribution according to the Fresnel zone plate law [2]. Flat polished (100) Si wafers with thickness 0.33 mm were used as substrates. To improve adhesion, the substrate surface was preliminary exposed to Ar<sup>+</sup> ions before deposition. The multilayers were deposited on the substrates at temperature  $T_s \approx 40^{\circ}$ C and on ones heated at  $T_s \approx 200^{\circ}$ C and  $T_s \approx 300^{\circ}$ C. The thickness of the layers in multilayers was defined by the exposure times of the substrate above the

| No.   | Туре        | Layers<br>materials   | Layer thickness,<br>nm | Periods<br>number/layers<br>number | <i>T<sub>s</sub></i> , °C | Multilayer<br>thickness, µm |
|-------|-------------|-----------------------|------------------------|------------------------------------|---------------------------|-----------------------------|
| 1     | Periodic    | TaSi <sub>2</sub> /Si | 25/25                  | 325/650                            | 40                        | 16                          |
| 2     | Periodic    | TaSi <sub>2</sub> /Si | 25/25                  | 325/650                            | 200                       | 16                          |
| 3     | Periodic    | TaSi <sub>2</sub> /Si | 25/25                  | 325/650                            | 300                       | 16                          |
| $4^*$ | Periodic    | W₅Si₃/Si              | 25/25                  | 325/650                            | 40                        | 16                          |
| 5     | Periodic    | TaSi <sub>2</sub> /Si | 10/25                  | 15/30                              | 200                       | 0.53                        |
| 6     | Periodic    | TaSi <sub>2</sub> /Si | 33/33                  | 10/20                              | 200                       | 0.66                        |
| 7     | Periodic    | TaSi <sub>2</sub> /Si | 25/25                  | 15/30                              | 200                       | 0.75                        |
| 8**   | Nonperiodic | TaSi <sub>2</sub> /Si | (25-30)/(25-30)        | /740                               | 200                       | 22                          |
| 9**   | Nonperiodic | TaSi <sub>2</sub> /Si | (25-30)/(25-30)        | /705                               | 200                       | 20                          |

\* — multilayer deposited in [2, 3]. \*\*— multilayer with depth graded layer thickness (25-33 nm)

corresponding target. One-hour annealings at 400°C and 510°C were carried out in a vacuum at a pressure below 0.003 Pa. The structure of layers and layer interfaces in multilayers were studied by transmission electron microscopy (TEM) in a microscope PEM-U operating at 100 kV. Preparation of multilayer cross-section included both mechanical and ion thinning. Small angle Xray diffraction (SAXRD) in  $Cu-K\alpha_1$  radiation ( $\lambda = 0.154051$  nm) was performed in DRON-3M diffractometer. SAXRD patterns were simulated using XRayCalc code [5] and Henke's atomic scattering factors [6]. Such approach allowed estimating the thickness, density of individual layers and interface roughness in multilayers. Computer simulation of SAXRD patterns was carried out for thin periodic TaSi<sub>2</sub>/Si multilayers with a small number of periods of 10-15. This was due to the minimization of the influence of the substrate bending on the shape of SAXRD pattern by mechanical stresses. Computer simulation of SAXRD patterns for multilayers with a number of periods of more than 15 made it possible to estimate the layer thickness and interface roughness only in the uppermost about 15 periods due to the absorption of X-ray in multilayer. Phase structure of layers of the multilayers was established with  $Cu{-}K\alpha$  radiation ( $\lambda=$ 0.154181 nm) by analyzing the  $\theta/2\theta$  X-ray diffraction patterns. The evaluation of the mechanical stresses in the system (TaSi<sub>2</sub>/Si multilayer)/Substrate was carried out according to the Stoney formula [7]:

$$\sigma = \frac{E_s h_s^2}{6(1 - v_s)h_f R_s},\tag{1}$$

with  $E_s$  as the Young's modulus of the substrate,  $h_s$  as the thickness of the substrate,  $v_s$  as the Poisson's ratio of the substrate,  $h_f$ as the multilayer thickness, and  $R_s$  as the curvature radius of the multilayer substrate. Values of  $R_s$  were measured using an X-ray diffractometer. The procedure for measuring  $R_s$  is described in [8].

#### 3. Results and discussion

We started with the TaSi<sub>2</sub>/Si multilayer deposited on a Si substrate at temperature of  $T_s \approx 40$  °C (Table, No. 1). The layer thickness of  $h_{\text{TaSi}_2} \approx h_{\text{Si}} \approx 25$  nm, total thickness of 16 µm TaSi<sub>2</sub>/Si multilayer was the same as in the case of W<sub>5</sub>Si<sub>3</sub>/Si multilayer (Table, No. 4) [2, 3]. The described design made it





Fig. 1. Cross-section TEM micrographs and selected area diffraction of the  $TaSi_2/Si$  multilayer, deposited at substrate temperature 200°C (Table, No. 7).

possible to compare mechanical stresses in both multilayers. The compressive mechanical stresses are in as deposited TaSi<sub>2</sub>/Si multilayer in the same manner as in the W<sub>5</sub>Si<sub>3</sub>/Si multilayer, which bent the substrate. The substrate bending only partially reduces a mechanical stresses in multilayer. The mechanical stresses in the TaSi<sub>2</sub>/Si multilayer and the W<sub>5</sub>Si<sub>3</sub>/Si multilayer (Table, No. 1, 4) have close values of  $\sigma \approx -440$  MPa and  $\sigma \approx -520$  MPa, respectively. Both multilayers, as we consider from the sign and the values of  $\sigma$ , have a common cause of mechanical stresses [7, 9] — the densification of the silicon layers by introduction of the Si atoms in this layers during deposition.

In order to find the optimum deposition temperature for the LZP with the minimum mechanical stresses, continuous layers and low interface roughness, TaSi<sub>2</sub>/Si multilayers were deposited on a heated substrates at  $T_s \approx 200^{\circ}$ C,  $T_s \approx 300^{\circ}$ C (Table, No. 2, 3). As expected, an increase in substrate temperature allowed reducing the mechanical stresses in multilayer. The mechanical stresses are  $\sigma \approx -320$  MPa (by factor of  $\approx 1.4$  less) at  $T_s \approx 200^{\circ}$ C, and  $\approx -270$  MPa (by factor of  $\approx 1.6$  less) at  $T_s \approx 300^{\circ}$ C (Table, No. 1, 2, 3). It was supposed, that decreasing the compressive mechanical



Fig. 2. Small angle X-ray diffraction patterns  $(\lambda \approx 0.154051 \text{ nm})$  of the TaSi<sub>2</sub>/Si multilayers with layers thickness  $h_{\text{TaSi}_2} \approx h_{\text{Si}} \approx 25 \text{ nm}$ , 16 µm multilayer thickness, deposited at substrate temperature 40°C (1), 200°C (2), 300°C (3) (Table, No. 1, 2, 3).

stresses in the multilayer with  $T_s \approx 300$  °C could be caused by the structure relaxation in Si layers [7, 9], beginning of structure change in TaSi<sub>2</sub> layers (see below) or tensile thermal stresses as a result of different TEC of layers and substrate.

The effect of the substrate temperature on the characteristics of growth of the TaSi₂/Si multilayers with $T_s \approx 40^{\circ} \mathrm{C},$  $T_s \approx 200^{\circ}$ C,  $T_s \approx 300^{\circ}$ C (Table, No. 1, 2, 3) was studied. In all three cases computer simulation of SAXRD patterns are well fitted by a two-layer ( $TaSi_2$  and Si) model without mixed layer. Fig. 1 shows a TEM cross section of the  ${\sf TaSi}_2/{\sf Si}$  multilayer (Table, No. 7) with  $T_s \approx 200^{\circ}$ C. This multilayer consists of continuous layers of tantalum silicide and silicon with sharp and smooth layers interface. The mixed zones on the layers interface are not detected. Based on the computer simulation results of SAXRD patterns from thin TaSi<sub>2</sub>/Si multilayers (Table, No. 5, 6, 7) with  $T_{s}\approx 200^{\circ}\mathrm{C},$ the density of 10 - 33 nm thick TaSi<sub>2</sub> layers and 25 - 33 nm thick Si layers is, respectively,  $\approx 8.6 \text{ g/cm}^3$  and 2.3 g/cm<sup>3</sup>. These values are consistent with the data ICDD Powder Diffraction File and [10], where the silicide TaSi<sub>2</sub> has a density of  $9.07 \text{ g/cm}^3$ and 8.83 g/cm<sup>3</sup>, and Si has 2.33 g/cm<sup>3</sup>. In the 16  $\mu$ m TaSi<sub>2</sub>/Si multilayer deposited at  $T_{s} \approx 200^{\circ}$ C (Table, No. 2) the interface boundaries are smooth with a constant interface roughness of  $\approx 0.3~\text{nm}$  for the overall multilayer depth. This is confirmed by the same interface roughness of thin



Fig. 3. X-ray diffraction patterns in Cu–K $\alpha$ radiation ( $\lambda \approx 0.154181$  nm) for the TaSi<sub>2</sub>/Si multilayers with layers thickness  $h_{\text{TaSi}_2} \approx h_{\text{Si}} \approx 25$  nm, 16  $\mu$ m multilayer thickness, deposited at substrate temperature 40° (1), 200°C (2), 300°C (3) (Table, No. 1, 2, 3) and for the TaSi<sub>2</sub>/Si multilayer (Table, No. 2) annealed at 400°C (4) and 510°C (5).

TaSi<sub>2</sub>/Si multilayers (Table, No. 7). In the 16  $\mu m$  thick TaSi\_2/Si multilayer with  $T_s \approx 40^{\circ} {
m C}$  (Table, No. 1) in the top layers, the interface roughness also is  $\approx 0.3$  nm. The diffraction peaks on the SAXRD pattern of the TaSi<sub>2</sub>/Si multilayer are present at the angles  $2\theta \approx (8 - 9)^{\circ}$  (Fig. 2). Due to low roughness, the  ${\rm TaSi}_2/{\rm Si}$  multilayers with  $T_s \approx 40^{\circ}$ C and  $T_s \approx 200^{\circ}$ C can be used for LZP with the thickness of individual layers from several nanometers and higher. With a further growth of the substrate temperature up to  $T_s \approx 300^{\circ}$ C, interface roughness increases in 16 µm TaSi2/Si multilayer (Table, No. 3). The interface roughness of the upper 30 layers is  $\approx 0.9$  nm according to the computer simulation results of SAXRD pattern. Diffraction peaks on SAXRD pattern of TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 300^{\circ}$ C are at angles less than  $2\theta \approx 4^{\circ}$ . Despite the increased roughness, such value of  $T_s$  is suitable for creating a multilayer for LZP with a layer thickness of 25-33 nm, since the roughness is less than 4 % of the thickness of the thinnest layers.

For a more complete description of the initial state and changes that occurred in the multilayer during thermal annealing, a study of the layer phase structure of the TaSi<sub>2</sub>/Si multilayer was made. For multilayers at  $T_s \approx 40^{\circ}$ C and  $T_s \approx 200^{\circ}$ C (Table, No. 1, 2) a TaSi<sub>2</sub> and Si layers are amorphous, there are 3 wide halos in X-ray diffraction

patterns (Fig. 3). The single diffraction peak near the angles  $2\theta \approx 69.4^{\circ}$  (Fig. 3) belongs to the (400) planes of the silicon single crystal substrate. Selected-area electron diffraction pattern for the TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 200^{\circ}$ C contains halos too (Fig. 1). The amorphous structure of the metal-silicide layers was also observed in other (W<sub>5</sub>Si<sub>3</sub>/Si, MoSi<sub>2</sub>/Si) multilayers [3, 11] and TaSi<sub>2</sub> film [12]. In the TaSi<sub>2</sub> layers of the TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 300^{\circ}$ C, the crystallization began — the peaks on the halos are slightly sharpened near the diffraction peak of tantalum disilicide (101), (111), and (203). Probably, the beginning of the layer crystallization in  $TaSi_2/Si$  multilayer with  $T_s \approx 300^{\circ}$ C is the reason of interface roughness growth up to  $\approx 0.9~\text{nm}$  indicated above.

As was shown above, a high value of mechanical stress is observed in the TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 200^{\circ}$ C. The mechanical stresses can lead, for example, to an uncontrolled separation of the multilayer from the substrate in the process of LZP manufacture by ionic and mechanical thinning [1]. To reduce the mechanical stresses the 16 μm TaSi<sub>2</sub>/Si multilayer (Table, No. 2) with  $T_s \approx 200^{\circ} \text{C}$  was annealed at  $T_s \approx 400^{\circ} \text{C}$ and  $T_s \approx 510^{\circ}$ C. After annealing at a temperature of 400°C the mechanical stress in the multilayer decreased by factor of 6.6 ( $\sigma \approx -48$  MPa). After annealing at 510°C, this multilayer crumbled. The magnitude and sign of mechanical stresses are mainly influenced, as we suppose, by the sum of two components that were related to the Si and the TaSi<sub>2</sub> layers. The first component, responsible for the compressive stresses is decreased with increasing annealing temperature due to partial structural relaxation in the Si layers [7, 9]. The second component responsible for tensile stresses increased after annealing because of structure changes in  $TaSi_2$  layer. Growth of tensile stresses occurred in  $TaSi_2$  film [12] on Si substrate after annealing at temperature  $\approx 450^\circ C$  and cooling to room temperature. Annealing temperature  $T \approx 450^{\circ}$ C is temperature of  $TaSi_2$  film crystallization [12].

The computer simulation of SAXRD patterns was carried out for of the TaSi<sub>2</sub>/Si multilayer annealed at  $T \approx 400$  °C and  $T \approx 510$  °C. Annealing of TaSi<sub>2</sub>/Si multilayer (Table, No. 7) deposited at  $T_s \approx 200$  °C is accompanied by changes in the multilayer construction. According to the results of the



Fig. 4. Period as function of annealing temperature for the  $TaSi_2/Si$  multilayer with layers thickness  $h_{TaSi_2} \approx h_{Si} \approx 25$  nm, 15 periods number, deposited at substrate temperature 200°C (Table, No. 7).

computer simulation, the best coincidence between the measured and fitted SAXRD pattern for TaSi<sub>2</sub>/Si multilayer annealed at  $T \approx 400^{\circ} \mathrm{C}$  is observed in the case of a slight decrease in the thickness of the TaSi<sub>2</sub> layer by  $\approx 0.1$  nm and an increase in the thickness of the Si layer by  $\approx 0.4$  nm. The density of the TaSi<sub>2</sub> layers remains constant at  $\approx 8.6$  g/cm<sup>3</sup>. The interface roughness did not change. An increase in the multilayer period (Fig. 4) due to structural relaxation in the Si layer was also observed in the annealed W<sub>5</sub>Si<sub>3</sub>/Si phase-nonequilibrium multilayer [3]. After annealing TaSi<sub>2</sub>/Si multilayer at  $T \approx 510^{\circ}$ C, the best coincidence to the measured SAXRD patterns occurs at the TaSi<sub>2</sub> layer thickness decreasing by  $\approx 0.3$  nm, the density increasing up to  $\approx 8.8 \text{ g/cm}^3$  and thickness of the Si layer decreasing by 0.5 nm. The increase in density perhaps occurs due to the crystallization of TaSi<sub>2</sub> layers. The estimation of the change in the  $TaSi_2$  layer thickness, is consistent with assumption based on the of volume change with increasing density at crystallization in this layer. After annealing at  $T \approx 510^{\circ}$ C, the interface roughness increases to  $\approx 0.4\,$  nm. In manufacturing of TaSi<sub>2</sub>/Si multilayer for LZP with subsequent annealing, these changes must be taken into account before the multilayer deposition in order to ensure maximum efficiency of the LZP.

As revealed by the studies of the layer phase structure, there is also no signs of crystallization in the TaSi<sub>2</sub>/Si multilayer (Table, No.2) deposited at  $T_s \approx 200^{\circ}$ C and annealed at  $T \approx 400^{\circ}$ C (Fig. 3). After an-

nealing at the temperature of  $510^{\circ}$ C, the multilayer diffraction pattern shows TaSi<sub>2</sub> crystallization (Fig. 3). The TaSi<sub>2</sub>/Si multilayer with layer thicknesses  $h_{Si} \approx h_{TaSi_2} \approx 33$  nm (Table, No. 6) annealed at  $T \approx 510^{\circ}$ C, and the TaSi<sub>2</sub>/Si multilayer with a set of layer thicknesses in the range of 25 - 33 nm (Table, No. 8) have the same diffraction patterns. Probably, the crystallization of the  $\mathsf{TaSi}_2$  layer is the cause in the interface roughness increase in thin TaSi<sub>2</sub>/Si multilayer (Table, No. 7) and change of sign of the mechanical stresses to the opposite (see below) and the crumbling of  $16 \ \mu m$ TaSi<sub>2</sub>/Si multilayer (Table, No. 2).

In this paper, we also investigated the possibility of deposition a TaSi<sub>2</sub>/Si multilayers with a thickness of more than  $16 \ \mu m$  to a substrate at a temperature  $T_s \approx 200^{\circ}$ C for the LZP at a wavelength of 0.1 nm with a focal length of  $\approx 20$  mm [2, 3]. The substrate temperature  $T_s$  was chosen to be equal to 200°C, since the TaSi<sub>2</sub>/Si multilayer has an optimal ratio of interface roughness and mechanical stress. Such multilayer can be used for LZP with a wide range of thicknesses from several nm up to tens of nm. The maximum thickness of the deposited TaSi<sub>2</sub>/Si multilayer was  $\approx 22 \ \mu m$  (Table, No. 8). This multilayer consisted of 740 layers of TaSi<sub>2</sub> and Si, with varying thickness close to the LZP (from 25 nm to 33 nm). The mechanical stress  $\sigma$  in the TaSi<sub>2</sub>/Si multilayer is about -350 MPa, which is close to  $\sigma$ in the one (Table 1, No. 2) with a thickness of 16  $\mu$ m and a  $T_s \approx 200$  °C. After annealing at  $T \approx 510^{\circ}$ C, part of these multilayer (Table, No. 8) separated from the substrate and partially peeled off, while the other part of the multilayer remained on the substrate without visible changes, bending the substrate in opposite direction. The tensile mechanical stress in a part of a multilayer without damage was  $\sigma \approx 125$  MPa.

It should be noted that  $20 - 22 \ \mu m$  was the maximum thickness of the multilayers for LZP. After depositing the multilayer of this thickness some of substrates had traces of splitting into the plates holding from separation by the multilayer, though some multilayer on substrate remained without visible damage. It can be assumed that the maximum thickness of the TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 300^{\circ}$ C could reach 24 – 26 µm according to the value of mechanical stress in 16 µm TaSi<sub>2</sub>/Si multilayer with  $T_s \approx 300^{\circ}$ C.



Fig. 5. Required (1) and estimated depth (2, 3) layer thickness distribution of  $T\dot{a}Si_2$  (3) and Si (2) from substrate to surface in 20  $\mu$ m TaSi<sub>2</sub>/Si multilayer, deposited at substrate temperature 200°C (Table, No. 9).

Due to the possible destruction of the multilayer mentioned above, the maximum thickness of the TaSi<sub>2</sub>/Si multilayer for LZP at deposition was limited by 20  $\mu$ m. After the fabrication of the TaSi<sub>2</sub>/Si multilayer (Table, No. 9) for the LZP, the thickness layers distribution (Fig. 5) along the depth was estimated from the thickness of the layers near the substrate and near the top of multilayer. An evaluation of the thickness of the layers near the substrate was carried out based on the simulation results of the SAXRD pattern of a preliminary deposited thin TaSi<sub>2</sub>/Si multilayer with a number of periods of 15. The thickness value of the top layers were obtained from the simulation of SAXRD pattern of TaSi<sub>2</sub>/Si multilayer with a thickness of 20 µm. Intermediate points on the graph were estimated by layers deposition time. As can be seen from Fig. 5, in multilayer the maximum deviation of the layer thickness distribution from the required one for LZP is observed in the near-surface layers, which were deposited last. This deviation thickness of the layers of  $TaSi_2$  and Si is, respectively, 0.45 nm and 0.15 nm. The difference between the required and total multilayer thickness is less than 10 nm. This means that the efficiency of the LZP would not change significantly, since none of the silicon layers that must transmit radiation in the LZP does not work in complete antiphase. The coincidence of layer thickness in such TaSi<sub>2</sub>/Si



Fig. 6. Scanning electron microscope image of linear zone plate based on the  $TaSi_2/Si$  multilayer with silicon substrate after ion etching.

multilayer is better than in  $W_5Si_3/Si$  multilayer [2, 3]. The introduction of an additional correction for the deposition rate drift, which is caused by the erosion of the magnetron target, would allow improve the coincidence of the obtained and the required LZP thickness distribution in multilayer.

The final stage of the work was the test fabrication of a LZP as rectangular stack from multilayer on a silicon substrate by Ar ion etching. The ion energy was 1.5 keV. The cross section of the LZP has a trapezoidal shape due to a mask width decrease by ion sputtering (Fig. 6). The height of the LZP is limited to 10  $\mu$ m due to the thinning and subsequent breakage of the wire mask.

#### 4. Conclusions

The features of the formation, structure, and mechanical stresses in TaSi<sub>2</sub>/Si multilayers deposited by magnetron sputtering on heated substrates in the initial state and after the thermal annealing were studied. Maximum thickness of TaSi<sub>2</sub>/Si multilayers deposited on Si substrates was  $20 - 22 \mu m$ . Formation of TaSi<sub>2</sub>/Si multilayers is accompanied by appearance of compressive mechanical stress. The mechanical stresses in the  $TaSi_2/Si$  multilayers was reduced from -440 MPa to -320 MPa and to -270 MPa with substrate temperature  $T_s$  increasing, respectively, from 40°C to 200°C and 300°C during deposition. The value of a mechanical stresses in  $TaSi_2/Si$  multilayer with  $T_s \approx 200^{\circ}$ C was reduced by factor of  $\approx 6.6$ 

after annealing at  $T \approx 400^{\circ}$ C. The crystallization of TaSi<sub>2</sub> layers occurs in TaSi<sub>2</sub>/Si multilayer after annealing at  $T \approx 510^{\circ}$ C. Annealing of the TaSi<sub>2</sub>/Si multilayer at  $T \approx 400^{\circ}$ C and  $T \approx 510^{\circ}$ C is accompanied by a change in the layers thickness and the whole multilayer thickness, which should be taken into account before multilayer deposition for linear zone plate. The TaSi<sub>2</sub>/Si multilayers with thickness 16  $\mu$ m and 22  $\mu$ m peeled of substrate after annealing at  $T \approx 510^{\circ}$ C. A TaSi<sub>2</sub>/Si multilayer with a thickness of 20 µm was fabricated with necessary depth layer distribution. Deviation of layer thickness is 0.45 nm for the TaSi<sub>2</sub> layer and 0.15 nm for the Si layer near the top of TaSi<sub>2</sub>/Si multilayer.

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