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Changes of the Defects Associated with He after the High-Temperature Annealing of Electronic Components with the Nanocrystalline Titanium Films Containing He

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Abstract: The annealing processing has a certain influence on the defect structure of the material. In this paper, titanium disc is taken as a cathode target with Si as the substrate. DC magnetron sputtering is used to prepare the nanocrystalline titanium film containing He used for electronic components of different concentrations. Then high temperature annealing treatment is carried out before observation and analysis. Finally, it's concluded that the electronic component takes use of the nanocrystalline titanium film that contains He to form TiSi2 crystal with preferential orientation. And the surface layer of sample titanium film and the defect structure of protective layer after annealing have changed. This study provides a useful reference for the research and application of the nanocrystalline titanium film containing He.

Keywords: Annealing treatment; Defect structure; Nanocrystalline titanium film containing He

1. Introduction

The He atoms in high concentration can enter the interior of material and cause changes in its properties. However, He is difficult to dissolve in the matrix of material, and the migration energy is very low. Therefore, He is more likely to migrate and diffuse, resulting in the generation of He bubbles, thereby increasing the internal defect of the material, accelerating the aging of materials and reducing the service life of materials [1]. At present, there are many studies on the influence of He bubble growth on material properties in materials at home and abroad, but the specific studies are not comprehensive [2]. In this paper, DC magnetron sputtering is used to prepare the nanocrystalline titanium film containing He used for electronic components of different concentrations, and the high temperature annealing treatment is carried out to study the change of He-related defects in the material.

2. Experimental Materials and Analytical Methods

In this paper, a titanium disk with a purity of 99.99% is used as the cathode target, and Si (100) is used as a substrate. DC magnetron sputtering is used to prepare the nanocrystalline titanium film containing He used for electronic components of different concentrations. The experimental environment is a He/Ar mixed atmosphere, and He/Ar flow ratio is taken as 8, 4, 2, and 0, respectively. Elastic recoil detection analysis is conducted by a tandem accelerator to measure He-containing titanium films at different concentrations under different He/Ar flow ratios (corresponding to obtain the concentrations of He are 14%, 5%, 2%, 1%, and 0% respectively). Then, take half of the sample for high temperature annealing treatment and then air cooled. Then, the microstructure of the sample after annealing treatment is measured by XRD diffractometer, and the grain size of He-containing titanium film and TiSi2 crystal before and after annealing are measured by Jade 5[3], measuring the slow positron-electron Doppler broadening of the material, and obtaining the S-parameter value of the He-doped titanium film by VEPFIT fitting.

3. Experimental Results and Analysis

3.1. Microstructure of He titanium film after annealing treatment

As shown in Figure1, it's the XRD spectral lines of the He titanium film doped at different concentrations after annealing treatment. It can be seen from Figure1 that the diffraction characteristic peaks of Ti crystal and Si crystal are not found in the XRD pattern of the sample after annealing. The software analysis shows that the diffraction peaks are consistent with the standard diffraction peaks of TiSi2 crystals, mainly because the titanium atoms in the titanium film react with the silicon atoms in the substrate in the interfacial layer after the annealing treatment of Ti/Si samples, resulting in interdiffuse and forming stable TiSi2 polycrystal, so it is not suitable to use Si as the substrate when preparing titanium film. The correspondence between the 2θ and the crystal plane of TiSi2 diffraction peak is: 39.1°-(311), 42.2°-(040), 43.2°-(022), and 49.7°-(331) respectively. The (022) peak in the titanium film without He is the strongest, and the preferred orientation of TiSi2 (022) crystal plane exists. When the He atom is introduced, the strongest peak becomes the diffraction peak of the (040) crystal plane, and the preferred orientation also becomes a TiSi2(040) crystal plane, which indicates that the introduction of He atoms can change the preferred orientation of TiSi2 crystal growth [4].

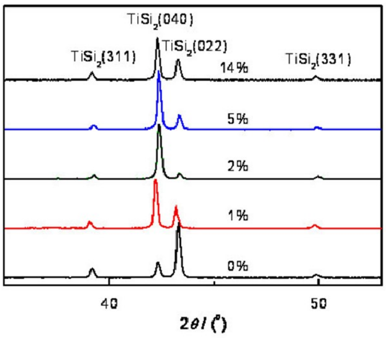


Figure 1. XRD spectra of He titanium films doped at different concentrations after annealing

As shown in Figure 2, it's the grain size of the unannealed and annealed He-doped nanocrystalline titanium film obtained by software analysis at different He concentrations. From Figure 2, it can be seen that the grain size of the sample which has not been annealed continuously decreases with the increase of He concentration, which indicates that He atom has an inhibitory effect on the nucleation growth of the nanocrystalline titanium film. The annealed samples fluctuate in a small range with increasing He concentration, but the amount of change is negligible, which indicates that the concentration of He atoms has no effect on the grain size of the annealed TiSi2 crystal.

3.2. Analysis of He-related defects of He-containing titanium film

The S parameter can reflect the momentum information of low momentum electrons, so it can be used to describe the structure and defects inside the material. As shown in Figure 3, the S-E curve of He-containing titanium film at different concentrations without annealing and after annealing treatment, and E is the implantation energy. It can be seen from Figure 3(a) that the surface of sample and the defects of Pd layer are positronically characterized when the energy is below 1 keV without annealing, and all the S-E curves are similar because the Pd of all the samples are the same, resulting in the S-E curve are relatively similar. The curve is basically horizontal between 2keV and 7keV, which indicates that the defect distribution in this range is relatively uniform [5]. When the He concentration is 14%, the S parameter is significantly increased, which indicates that the number of vacancy type defects is greatly increased.

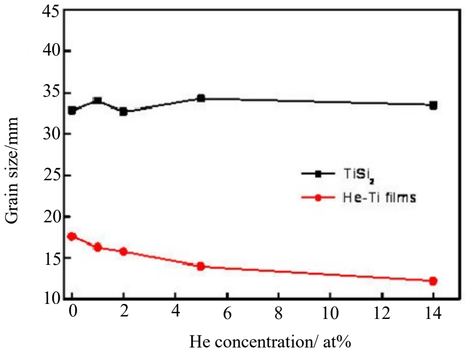
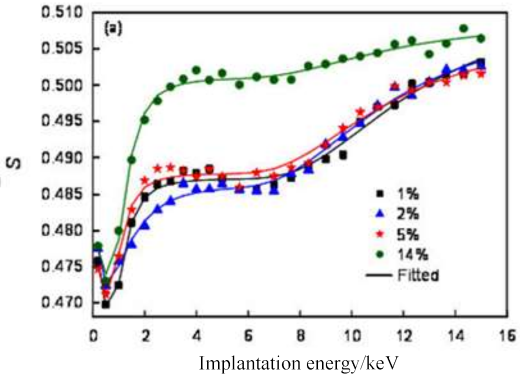
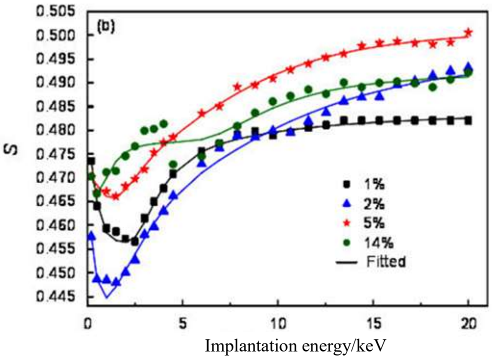


Figure 2. Grain size of He-doped nanocrystalline titanium film without annealing and after annealing at different He concentrations

From Figure 3(b), it is found that the surface structure of sample after annealing and the defect structure of the protective layer are changed, resulting in a large difference in the energy required for the positron of the Pd-Ti interface layer, so the difference of SE curve is bigger. The appearance of horizontal portion is not observed in the S-E curve of the sample titanium film after annealing, which indicates that the annealing causes the distribution of defects in the range to be no longer uniform. When the implantation energy exceeds 5 keV, the S parameter value of sample titanium film having a He concentration of 5% is larger than that of the sample having a He concentration of 14%.



(a) Without annealing treatment



(b) After annealing

Figure 3. S-E curve of He-containing titanium film with different concentrations without annealing and after annealing treatment.Without annealing and after annealing at different concentrations of titanium-containing SE graph He films

As shown in Figure 4, the S-parameter fitting value of He-containing titanium film without annealing and after annealing treatment is varied with concentration. It can be found that the S value of titanium film of the sample after annealing is reduced, which is mainly because the annealing treatment eliminates the vacancy type defects generated during the preparation of the titanium film, and the TiSi2 crystal formed by annealing also has a certain influence on the S parameter. The S parameter value of titanium film of the unannealed sample is increased when the concentration of He is 5% or less, which is mainly caused by the increasing of defect concentration. When the concentration is within the range of 5% to 14%, the S value increases significantly with increasing concentration, which is mainly caused by the increasing of the concentration and size of He-related defects. The S value of titanium film after annealing treatment is significantly larger in the range of He concentration from 0% to 1% and from 2% to 5%, and approximately unchanged in the range from 1% to 2%, which indicates the concentration of He has a great influence on the internal defects of titanium film. The S value is significantly reduced in the range from 5% to 14%, which may be due to the fact that the degree of decrease of He-related defect concentration in the high He concentration titanium film sample is much larger than the size enlargement degree of He bubble defect due to the annealing treatment, thereby causing S parameter value of the characterization vacancy defect reduced.

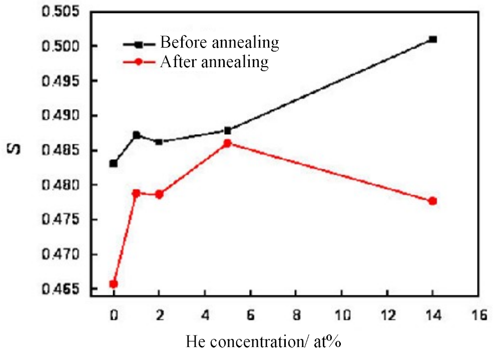


Figure 4. Variation of S-parameter fitting value of He-containing titanium film without annealing and after annealing treatment

4. Conclusion

In summary, the TiSi2 crystal having a preferential orientation is formed in the He-containing nanocrystalline titanium film for the electronic component, and the introduction of He atom can change the preferred orientation of TiSi2 crystal growth. He atoms have an inhibitory effect on the nucleation growth of nanocrystalline titanium film, and the concentration of He atoms has no effect on the grain size of the annealed TiSi2 crystal. The surface structure of the sample after annealing and the defect structure of the protective layer are changed. After the annealing treatment, S value of titanium film of the sample is reduced, and the annealing treatment eliminates the vacancy defect generated during the preparation of titanium film.

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