IRRADIATION-INDUCED NANOSTRUCTURES

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This paper summarizes the results of our studies of the irradiation-induced formation of nanostructures, where the injected interstitials from the source of irradiation are not major components of the nanophase. This phenomena has been observed by *in situ* transmission electron microscopy (TEM) in a number of intermetallic compounds and ceramics during high-energy electron or ion irradiations when the ions completely penetrate through the specimen. Beginning with single crystals, electron or ion irradiation in a certain temperature range may result in nanostructures composed of amorphous domains and nanocrystals with either the original composition and crystal structure or new nanophases formed by decomposition of the target material. The phenomenon has also been observed in natural materials which have suffered irradiation from the decay of constituent radioactive elements and in nuclear reactor fuels which have been irradiated by fission neutrons and other fission

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products. The mechanisms involved in the process of this nanophase formation are discussed in terms of the evolution of displacement cascades, radiation-induced defect accumulation, radiation-induced segregation and phase decomposition, as well as the competition between irradiation-induced amorphization and recrystallization.

Introduction

Nanocrystalline materials are generally defined as single-phase or multi-phase polycrystals of ultra-fine grain sizes (1 to 25 nm), and they may consist of crystalline, quasicrystalline or amorphous phases [1-3]. Nanocrystalline materials can be classified into several categories according to the nanostructure dimensionality [4], i.e., zero-dimensional atomic clusters and cluster assemblies, one-dimensional nano-tubes, two-dimensional modulated multilayers and three-dimensional equiaxed nanostructures. A great deal of effort has gone into the synthesis and characterization of the three-dimensional nanocrystalline materials because they often have properties superior to those of the conventional coarse-grained polycrystalline materials [3,5]. For example, a material with metal nanoparticles in dielectrics has recently become one of the most promising candidates for photonic materials because its unique optical nonlinearity and rapid response [6,7]. Also, nanocrystalline ceramics have been demonstrated to have improved ductility [8] and even potential superplasticity [9]. Among the commonly known techniques used to synthesize nanophase materials are *in situ* consolidation of the ultrafine particles, mechanical attrition, spray conversion processing, severe plastic deformation, sputtering, electro-deposition, rapid quenching and complete crystallization of amorphous solids [5]. In recent years, ion beam technologies have increasingly been used as an effective tool for creating nanostructured materials [10-12]. However, most of the application of ion beam techniques have been

focused on the formation of nanocrystals by ion implantation with doses above the solubility limit of the host material followed by thermal annealing. The nanocrystals formed by this method are either entirely or partially composed of the implanted species. Although the phenomenon of irradiation-induced polycrystallization was reported earlier for materials like MgO [13] and UO₂ [14], there has been very limited study in the nanometer scale on this subject.

In this paper, we present a summary of our results on studies of irradiation-induced three-dimensional nanophase formation, in which the concentration of the implanted species from the source of radiation are so low that they do not constitute major components of the nanophases, or rather, the nanophases are induced solely through the beam-solid interactions. Beginning with single crystal or coarse-grained polycrystalline materials, irradiation with energetic particle beams may result in nanostructures composed of either nanocrystals of the original composition and structure or nano-scaled new phases. This phenomenon has been reported earlier by Wang et al. [15] for a number of intermetallic compounds and ceramic materials based on observations with *in situ* transmission electron microscopy (TEM) during studies on radiation-induced amorphization [16], and it was mostly evident in materials which are susceptible to solid-state amorphization near the critical amorphization temperature at which amorphization and crystallization are in a close competition. At the present time, the number of materials studied has increased. A similar phenomenon has also been observed in naturally occurring materials which have suffered radiation from the decay of constituent radioactive elements [17], as well as in nuclear reactor fuels after high burn-up [14]. The mechanisms for the process of this type of nanophase formation are related to the evolution of displacement cascades, radiation induced defect migration and accumulation,

radiation-induced segregation and phase decomposition. A better understanding of the controlling mechanisms for the process of irradiation-induced nanophase formation may lead to the development of new technologies for creating nanophase materials for engineering applications.

2. Experimental Procedures

The materials in which irradiation-induced nanophase formation have been observed include intermetallic compounds, such as U_3Si and Zr_3Al , and many natural and synthetic ceramic materials, such as $ZrSiO_4$ (zircon), Mg_2SiO_4 (olivine), $\gamma\text{-Fe}_2SiO_4$ (a high-pressure spinel phase), $(Pb_{0.91}La_{0.09})(Zr_{0.65}Ti_{0.35})O_3$ (PLZT 9/65/35), $Ca_5(PO_4)_3(F,OH,Cl)$ (F-apatite), $Ca_2La_8(SiO_4)_6O_2$ (apatite), Tl-1212 and Tl-2212 Tl-Ba-Ca-Cu-O high- T_c superconductors and UO_2 . The samples were either single crystals or polycrystalline aggregates with grain sizes in the order of tens of microns so that they could be studied as single crystals under TEM. TEM samples were prepared by mechanical polishing followed by 3-5 kV Ar ion milling which did not cause observable damage under high resolution TEM (HRTEM).

Ion irradiations were conducted at various temperatures with 500 keV to 1.5 MeV Kr^+ or Xe^+ ions using the HVEM-Tandem Facility at the Argonne National Laboratory [18]. The facility consists of a modified Kratos/AEI high voltage electron microscope (HVEM) which is interfaced to a 2 MV tandem ion accelerator, so electron diffraction and TEM images can be monitored *in situ* during irradiations. A 300 keV electron beam was used for the *in situ* TEM observation. Because of the high ion energy and small specimen thickness (<300 nm) in the region of observation, most ions penetrate through the sample, and the amount of implanted ions is negligible as estimated by Monte Carlo calculations with the TRIM code [19]. The temperature of the sample stage was controlled and monitored during irradiations. Additional detailed TEM or HRTEM examination of the irradiated samples were conducted

after various ion doses. The effects of electron beam irradiation were studied in the transmission electron microscopes with electron energies ranging from 200 keV to 1 MeV. The HRTEM was mostly performed with a JEM 2010 electron microscope operating at 200 kV. In addition, the microstructure of naturally occurring samples damaged by alpha-decay events of the constituent radionuclides have also been studied with TEM.

3. Results and discussion

As an energetic ion traverses a target, it loses its energy predominantly through electronic (ionization) and nuclear (elastic collision) interactions with the target atoms. The target atom which receives sufficient energy through elastic collision may be displaced from its lattice site and may further collide with and displace other target atoms, thus creating a displacement cascade. Single cascade damage formation occurs within 10 ps in most metals according to molecular dynamic calculations [20]. Depending on the energy and mass of the incident ions and the target material, the size of a displacement cascade can be from a few nanometers to a few hundred nanometers. Molecular dynamic calculations have also shown that the cascade volume is liquid-like when it is first created. If the cooling rate of this volume is sufficiently high, the cascade can be directly quenched into a nanometer-scaled amorphous domain, otherwise it anneals quickly with some point defects or defect clusters remaining. Complete amorphization can be achieved by the gradual accumulation of the amorphous domains (which can also be created by cascade overlap) or through the accumulation of point defects. Amorphization can be induced by irradiation of most complex ceramics and many intermetallic compounds, but not in most metals and many simple oxides. The dose required for complete amorphization of an irradiated material usually increases with the increasing temperature due to the increased competition from crystallization processes [21].

The materials listed in this study can most easily be amorphized at room temperature

under heavy ion irradiation at a fraction of a displacement per atom (dpa) [16,22-27] except for UO_2 . In most cases, radiation-induced nanophase formation has been found to be closely related to the process of radiation-induced solid-state amorphization.

3.1 Radiation-induced formation of nanocrystals of the original phase

Nanocrystals of the same type as the original phase in an amorphous matrix can be formed by the process of radiation-induced amorphization of many complex ceramic materials in which the development and accumulation of amorphous domains are probably caused by direct cascade quenching or cascade overlap and thus are inhomogeneous in character. An example is given by HRTEM images taken of synthetic zircon after various doses of 1.5 MeV Kr ion irradiation at the room temperature (Figs. 1). During irradiation, the microstructure first changed from the single crystalline state (Fig. 1(a)) to a microstructure with isolated amorphous domains in a single crystal matrix (Fig. 1(b)). The size of these amorphous domains (2-10 nm) are of the same order as that of displacement cascades in the thin specimen, indicating that amorphization is through either direct cascade quenching or cascade overlap. With an increase of irradiation dose, the volume fraction of the amorphous domains increased and the microstructure gradually changed into that of a nanophase material which has nanocrystals (~ 3 nm) in an amorphous matrix (Figs. 1(c)). Similar results have also been observed in Kr^+ ion irradiated olivine [15]. Although the nanocrystals are “the left overs” of the original single crystal, they showed a certain degree of misorientation or rotation (less than ± 10 degrees) (Fig. 2(c)) due to the locally high, anisotropic strain field caused by the volume expansion of the amorphous domains. The slight rotation of the remaining crystallites is often represented in the electron diffraction

pattern as an arcing of the diffraction spots as seen in textured polycrystalline materials.

Similar effects have also been observed in naturally damaged materials after various doses of self-irradiation from the alpha-decay events [17,25,28]. Based on the changes in x-ray diffraction and electron diffraction patterns, as well as observations made by HRTEM, Lumpkin and Ewing [28] and Murakami et al. [17] have proposed three stages in the process of alpha-decay induced amorphization with increasing alpha-decay dose. Stage I is characterized by Bragg powder diffraction maxima that decrease in intensity by a factor of two but remain sharp and the diffuse-scattering component is several orders of magnitude less than the Bragg diffraction maximum. Positions of diffraction maxima shift to lower values of 2θ that correspond to significant increase in the unit-cell volume. Also at this stage, The HRTEM shows a characteristic mottled contrast and isolated lattice-fringe free domains of 1-5 nm in size. At stage II, the Bragg diffraction maxima broaden and significantly decrease in intensity with the significant increase of the amorphous fraction. Lumpkin and Ewing [28] have pointed out that the decrease in grain size is one of the most important features of stage II, and the small periodic domains in this stage can have severe distortions. At stage III, the material is x-ray and electron diffraction amorphous. The nanocrystalline features illustrated in Fig. 1(c) evidently fall into stage II of the amorphization process.

Clearly, nanocrystals formed by the above mechanism will have a preferred orientation (the original orientation), although they may rotate slightly. Microstructures with randomly oriented nanocrystals have also been observed in self-irradiated minerals, as exemplified by the HRTEM image and electron diffraction pattern from a Sri Lankan zircon specimen in Fig. 3. Another major difference between the features in Fig. 3 and the stage II of the

amorphization process is that the nanocrystals in Fig. 3 almost completely occupy the entire sample volume instead of being well isolated in an amorphous matrix as shown in Fig. 1(a). The microstructure is similar to that obtained from the crystallization of the amorphous material [5] and, indeed, it could be the result of recrystallization associated with thermal events in the sample history. Nevertheless, similar results have been obtained in several intermetallic and ceramic materials (such as U_3Si , Zr_3Al and $\text{Ca}_2\text{La}_8(\text{SiO}_4)_6\text{O}_2$) under ion beam irradiation in a well controlled thermal environment. This demonstrates a process which is different from classical crystallization of amorphous materials by thermal annealing [15,27, 29].

The original microstructure of the U_3Si and Zr_3Al before irradiation consisted of large grains tens micrometers in size and large martensite plates. At room temperature, the critical doses for complete amorphization are $1.2 \times 10^{18} \text{ Kr}^+/\text{m}^2$ for U_3Si (0.3 dpa) and $2.7 \times 10^{18} \text{ Kr}^+/\text{m}^2$ for Zr_3Al (0.8 dpa). The critical dose increases with the increasing irradiation temperature and the upper temperature limit for amorphization of U_3Si and Zr_3Al is $\sim 300^\circ\text{C}$ and 250°C , respectively [29]. During irradiation above these critical temperatures (390 - 450°C), the contrast of Martensite plates quickly disappeared from the TEM image, and small grains of less than 20 nm in size were observed below a dose of $2.7 \times 10^{19} \text{ Kr}^+/\text{m}^2$ for both materials. The microstructures of U_3Si before and after irradiation to this dose at 390°C are shown in Fig. 3. The nanocrystalline grains increased in number and appeared to increase very slowly in size during continued irradiation. Deterioration of the initial single crystal diffraction maxima was concurrent with formation and strengthening of the polycrystalline rings in the electron diffraction pattern. At a dose of $1 \times 10^{20} \text{ ions}/\text{cm}^2$, both

samples were almost completely transformed into polycrystalline materials with grains of ~20 nm in dimension. As indicated by the arcs in the original diffraction spots at early stages of irradiation, the small crystallites initially exhibited a degree of alignment with the original crystal orientation. This preference has been observed by HRTEM of a Zr_3Al sample irradiated at 450°C [15] which reveals that nanocrystallites (< 5 nm in dimension) are twisted relative to one another but are within a ~20° range. The crystallites may have been parts of the original crystal, but were separated by heavily damaged regions. However, new crystallites with a random orientation must have nucleated during continued irradiation. Because the irradiation was conducted well above the critical temperature for amorphization, the nucleation of the nanocrystals is expected to be directly from the crystalline state of the original specimen. One interesting property of these nanocrystalline materials is that they are resistant to radiation-induced amorphization even at temperatures below the critical temperature for amorphizing the bulk materials [29].

A three-dimensional nanocrystalline material has also been obtained by 1.5 MeV Kr^+ irradiation of a single crystal of $Ca_2La_8(SiO_4)_6O_2$ to 1×10^{18} ions/m² at 400°C [27] as shown in Fig. 4. The nanocrystals have been confirmed by electron diffraction to have the same crystal structure as the original phase. The irradiation temperature is 40°C below the critical amorphization temperature for the 1.5 MeV Kr^+ ions; however, the ionization or subthreshold interactions from the 300 keV electrons may shift the critical temperature to slightly lower temperatures. Thus, the nanocrystal formation and complete polygonization in irradiated $Ca_2La_8(SiO_4)_6O_2$ is apparently a result of the competition between amorphization and recrystallization that may have been enhanced by the simultaneous 300 keV electron-

beam irradiation. The literature provides evidence that electron-irradiation may induce or enhance crystallization of amorphous materials [27,30]. The process may involve radiation-enhanced diffusion, but certainly is not mainly due to the temperature increase from the electron beam heating [30].

The irradiation-induced grain size refinement has been noted to occur at the outer shell of the UO_2 nuclear reactor fuel after extended burn-up and is generally termed the "rim effect" [14]. In the outer shell of 150-200 μm thickness, the burn-up is increased by up to a factor of 2.5 due to neutron resonance capture by U-238 forming fissile Pu-239. The consequence is a grain-subdivision process or polygonization, i.e., the grains of the as-sintered UO_2 of typically 10 μm diameter are subdivided into some 10^4 new small subgrains of 0.2 to 0.3 μm size. Ion irradiation and electron microscopy have been used to study this process [31]. It was found that with 500 keV Xe^+ irradiation to 5×10^{15} ions/ cm^2 (20 dpa) at 170°C (including the beam heating) the dislocation density in the irradiated UO_2 is at least an order of magnitude higher than in the original sample. HRTEM has indeed revealed many sub-grain boundaries across which crystals are slightly rotated (1-2 degrees) with respect to one another. Also, after a simple filtering of the HRTEM images in Fourier space [31], it was evident that the sub-grain boundaries are associated with the ends of edge of dislocations (Fig. 5). This seems to be the initiating point of the subgrain formation. Either some of these subgrains develop into individual grains with larger rotation angle or the the increased strain field caused by increased dislocation density will eventually lead to recrystallization at the elevated temperatures, just as in the cold-working of metals. The latter may be a more plausible mechanism for irradiation-induced recrystallization.

Recrystallization from both the crystalline and the amorphous states can be achieved at much lower temperatures with energetic beam irradiations. One of the examples is that of partially amorphized PLZT 9/65/35 irradiated at 450°C [15]. The resulting microstructure contains crystallites of 10 to 100 nm distributed in an amorphous matrix and with small cavities of 10 to 40 nm in dimension attached to the edges of the crystallites. This microstructure is similar to that of an annealed amorphous material, although completely amorphized PLZT did not crystallize during *in situ* annealing in the microscope until above 550°C within the time frame of the irradiations. The formation of cavities is the result from the large density difference between amorphous and crystalline PLZT and supersaturation of vacancies generated by the irradiation.

The fact that irradiation-induced nano-scale polycrystallization occurs at temperatures near the critical amorphization temperature but below normal recrystallization temperature suggests that this is a result of the competition between amorphization and thermally-activated structural recovery. The observed stability of the small grain size against grain growth requires further investigation.

3.2 Radiation-induced formation of nanocrystals by chemical decomposition

In several multi-cation ceramic materials, radiation-induced formation of nanocrystals with compositions and structures different from the original host material have been observed [26,32-35]. Although the major mechanisms responsible for this process may include radiation-induced chemical segregation and decomposition, several unique material-related features have been noted in individual cases.

An example for radiation-induced decomposition of a metastable phase is given by the

irradiation of $\gamma\text{-Fe}_2\text{SiO}_4$, a high-pressure phase with the spinel structure. Although the Mg_2AlO_4 spinel is known as one of the most "amorphization-resistant" ceramic materials, $\gamma\text{-Fe}_2\text{SiO}_4$ is even less "resistant" to amorphization than its olivine polymorph, fayalite ($\alpha\text{-Fe}_2\text{SiO}_4$) [33]. After a moderate dose (2.9×10^{14} ions/cm²) of 1.5 MeV Kr^+ irradiation at 600°C, $\gamma\text{-Fe}_2\text{SiO}_4$ has been observed to decompose into nanocrystalline magnetite ($\text{Fe}^{2+}\text{Fe}^{3+}_2\text{O}_4$), an inverse spinel, and quartz (SiO_2) in an amorphous matrix as shown in Fig. 6. The grain size of these two decomposed phases increases with increased electron dose during TEM observation. The identity of the two decomposed phases has been confirmed by both convergent beam electron diffraction (CBED) and nano-beam energy dispersive x-ray spectroscopy (EDS) analysis. The irradiation temperature is below the critical amorphization temperature for quartz for 1.5 MeV Kr^+ irradiations; therefore, the growth of quartz crystallites may have been induced by the electron beam during TEM observation after the Kr^+ beam ceased. The decomposition of amorphous $\gamma\text{-Fe}_2\text{SiO}_4$ at elevated temperature is expected, as it is unlikely that the $\gamma\text{-Fe}_2\text{SiO}_4$ will be nucleated from the amorphous matrix during irradiation at elevated temperatures because it is far outside of its P-T stability field. For $\gamma\text{-Fe}_2\text{SiO}_4$ radiation-induced amorphization and decomposition must be much enhanced due to its thermodynamic instability.

Radiation-induced decomposition into component oxides has also been observed in tetragonal zircon and hafnon (HfSiO_2) (which is isostructural with zircon) under 800 keV Kr^+ irradiation at elevated temperatures [34,35]. Under irradiation at 600°C, zircon first becomes amorphous, but with increased ion dose, it gradually decomposes into nanocrystalline cubic or tetragonal ZrO_2 and amorphous SiO_2 . At temperatures above 750°C, the single crystal

zircon decomposed directly into the component oxides without an intermediate amorphous stage. The resulting microstructure is composed of randomly oriented nanometer-scale grains of ZrO_2 in the matrix of amorphous SiO_2 , as shown in Fig. 7. Similarly, nanocrystalline HfO_2 was observed in hafnion irradiated at elevated temperatures. Because both zircon and hafnion are stable phases in the temperature range over which the irradiations were conducted, the interpretation of the decomposition mechanism is not as straightforward, as it is in the case of $\gamma\text{-Fe}_2\text{SiO}_4$. Meldrum et al. [35] has proposed a convincing mechanism for the process which is based on cascade melting and quenching. Molecular dynamic simulations on the displacement cascade have suggested that during the thermal spike of the cascade formation, the temperature in the cascade core can be extremely high and atoms inside the displacement cascade may achieve a "liquid-like state". The phase diagram for the $\text{ZrO}_2\text{-SiO}_2$ system reveals that crystalline ZrO_2 coexists with a SiO_2 -rich liquid in the temperature range between 1960 and 2670 K for the composition of ZrSiO_4 . When the irradiation temperature is low, the cooling rate of the "liquid-like" cascade may be high enough to allow direct quenching into the amorphous state, as recently shown by both experimental results and theoretical analysis by Wang et al. for the $\text{MgO-Al}_2\text{O}_3\text{-SiO}_2$ system [36]. However, a lower cooling rate due to a much increased irradiation temperature may allow nucleation of ZrO_2 in the cascades. The size of the ZrO_2 crystallites formed by this mechanism is limited by the size of displacement cascades.

Another example supporting the mechanism of cascade quenching in nanocrystal formation through decomposition has been observed by Newcomer et al. [26] in a study of radiation effects in Tl-rich high- T_c superconductors in a temperature range between 20 and

673 K. Complete amorphization of Tl-1212 and Tl-2212 superconductors was achieved only in a temperature range between ~90 to 450 K under 1.5 MeV Kr⁺ and Xe⁺ irradiations. Nanocrystalline Tl₂O in an amorphous matrix was observed after irradiation at both 22 K and 470 K. Complete amorphization was expected at 22 K based on both experience and the assumption that crystallization would be suppressed at the low temperature due to the lack of atomic mobility. However, based on a careful consideration of the temperature dependence of the thermal conductivity of the material, Newcomer et al. [26] has described a new mechanism which controls the nano-phase formation. The thermal conductivity reaches its highest value at around 90 K, the upper limit for the superconductivity, and drops dramatically below this temperature. The cooling rate of the thermal spike of the cascade is directly related to the thermal conductivity of the surrounding material, that is a high thermal conductivity corresponds to a high cooling rate and vice versa. Thus, it is reasonable to expect to have the minimum amorphization dose at 90 K because it corresponds to the highest quench rate for the cascades. The cooling rates of a cascade cooling from the melting temperature of the material at various ambient temperatures have been estimated using a one dimensional classical heat transfer calculation. The calculation has resulted in very similar cooling rates for the ambient temperatures of 22 K and 500 K based on the fact that thermal conductivity is a factor of 2.5 smaller than that at 500 K (otherwise, the cooling rate of the cascade at the lower ambient temperature should be higher). This explains the similar results for the formation of nanocrystals of Tl₂O at both 22 K and 470 K.

Finally, we emphasize that nanophase formation through chemical decomposition can be induced by either collisional irradiation (as in the case of heavy ion irradiations) or ionizing irradiation (as in the case of electron irradiations). As shown in Fig. 8, nano-scaled cavities

and nanocrystalline CaO have been induced by 200 keV electron irradiations of hexagonal fluor-apatite $[\text{Ca}_5(\text{PO}_4)_3(\text{F},\text{OH},\text{Cl})]$ in the electron microscope. The presence of nanocrystalline CaO is indicated by both diffraction rings and Moiré fringes developed in the electron diffraction pattern and the TEM image obtained during the course of the irradiation [32]. The ionizing radiation breaks the weak bonds in the apatite structure through electronic excitation or a radiolysis process, causing the loss of volatile elements in the structure, such as F, Cl and even P. This results in the formation of cavities and the precipitation of CaO nanocrystals. A recent study has indicated that a high dose rate is essential to this process [30]. Experiments completed to date have not revealed any significant instability of CaO under further electron irradiation. Both collisional and ionizing radiation effects can also be used to promote the precipitation of implanted species at relatively low annealing temperatures in ion-implanted samples through radiation-enhanced diffusion and chemical segregation.

Conclusions

Nanocrystalline materials can be obtained through irradiation of coarse-grained intermetallic or ceramic materials with energetic particles without the incorporation of the implanted species. By carefully controlling the experimental conditions, nanostructures composed of either nanocrystals in an amorphous matrix or only nanocrystallites have been achieved. The irradiation-induced nanocrystals can be either the original phase of the target material or phases with different compositions and crystal structures. The process may involve radiation-induced partial amorphization, radiation induced- or enhanced-recrystallization and radiation induced-phase decomposition, through mechanisms such as cascade quenching, defect accumulation and radiation-induced segregation. These effects can be achieved by either collisional or ionizing irradiation or a combination of both.

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Figure Captions

Fig. 1. HRTEM images of synthetic ZrSiO_4 (zircon) taken before (a) and after 1.5 MeV Kr^+ irradiation to 1.7 (b) and 5.1×10^{17} ions/ m^2 (c) at the room temperature showing the creation anocrystalline zircon in an amorphous matrix by irradiation.

Fig. 2. HRTEM micrograph and selected area electron diffraction pattern of zircon from Sri Lanka (sample GRR 340). The sample has suffered irradiation from the alpha-decay of the constituent radionulides (mainly uranium) and is composed of randomly oriented nanocrystalline zircon particles with an average diameter of ~ 10 nm.

Fig. 3. Bright-field TEM micrograph of U_3Si taken *in situ* during irradiation by 1.5 MeV Kr^+ at 390°C ; (a) before ion irradiation, (b) after 2.7×10^{19} ions/ m^2 .

Fig. 4. HRTEM micrograph of a $\text{Ca}_2\text{La}_8(\text{SiO}_4)_6\text{O}_2$ specimen before (a) and after 1.5 MeV Kr^+ irradiation to 1×10^{18} ions/ m^2 at 400°C (b). The thin region of the sample has become nanocrystalline due to the irradiation.

Fig. 5. Filtered HRTEM micrographs showing the (111) lattice fringes of a UO_2 specimen before (a) and after 500 keV Xe^+ irradiation to 1×10^{20} ions/ cm^2 (b) at 170°C .

Fig. 6. Decomposition and formation of nanocrystals induced by 1.5 MeV Kr^+ irradiation in $\gamma\text{-Fe}_2\text{SiO}_4$ (a high-pressure spinel) at 600°C .

Fig. 7. HRTEM image of zircon (ZrSiO_4) irradiated by 800 keV Kr^+ at 775°C to 3 dpa showing the formation of randomly oriented ZrO_2 nanocrystals (highlighted) in amorphous SiO_2 matrix.

Fig. 8. HRTEM images and electron diffraction patterns of F-apatite taken before (a) and after (b) ten minutes of 200 keV electron irradiation showing the formation of nanocrystalline CaO (manifested by the Moiré fringes) and nano-scaled cavities.

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Fig. 3. The ring pattern of electron diffraction also suggests a zircon structure of the nanocrystalline phase.

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