## EMANATION THERMAL ANALYSIS USED TO STUDY ANNEALING OF RADIATION DISORDER

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## ABSTRACT

The possibilities of using emanation thermal analysis in the study of annealing of radiation defects are demonstrated and the prospects of the method in this field are outlined.

In this contribution, we draw attention to the not yet fully exploited possibility of using ETA (i.e., radioactive inert gas release measurements) in studies of the annealing of radiation disorder and to review some results obtained in this field so far.

It is well known that two types of disorder or damage are produced when energetic particles impact on a solid. The first is connected with ionization and electronic excitation and consists in the transfer of electrons in the solid and their trapping in quasi-stable positions. From these positions electrons are released during thermal annealing when the solid is heated.

The second type of disorder is induced particularly by bombardment with heavy particles and consists in displacement of atoms from their normal lattice positions and the formation of interstitials, vacancies and other lattice defects. Large doses of irradiation lead, through accumulation of these defects, to disordering and with certain types of solids to their conversion into an amorphous state. This type of disorder is also unstable, and the solid recrystallizes when heated and we shall focus our attention on the applications of ETA in studies of this amorphous crystalline transition.

It is interesting that this type of disorder and its annealing were first noted with certain minerals containing radioactive elements (metamict minerals). Here amorphization was due to bombardment by alpha recoil atoms over long geological periods. Release of the energy stored in the disordered lattice could be measured by DTA [1]. In the past two decades, lattice disordering by low-energy ion bombardment has acquired considerable importance, especially in connection with ion implantation of semicon-

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ductors. The lattice disorder thus produced and its annealing can be studied by various methods. Valuable information on various stages of disorder annealing can also be obtained using various ETA techniques. Some time ago we were involved in annealing studies of the disorder created by ion bombardment using the release of <sup>85</sup>Kr as a marker [2]. Such an application is made possible by the general phenomenon that individual stages of lattice recovery are accompanied by the release of inert gas atoms trapped in the solid.

The particular ETA technique that is appropriate for use in individual cases depends on the spatial distribution of the disorder in the solid. When the disorder is present in the whole volume of the solid, the classical emanation method with radon or thoron supplied from internally incorporated parent nuclides would be suitable. Here also, the technique based on the measurement of the release of the inert gases that had been generated within the solid using a neutron-induced nuclear reaction can be used. The release of the radioactive inert gas can be measured integrally by determining the fraction of the gas activity released during various annealing stages or measuring the fraction not released. Another convenient method for these purposes consists in the differential measurement of the sample. In this case peaks in the radioactivity release rate are recorded, which can be identified with the individual annealing stages.

A system of classification of various stages of inert gas release by correlation with disorder annealing stages has been suggested by Matzke [3]. A very distinct gas release stage accompanies particularly the amorphous-crystalline transition in solids that were converted into the amorphous state by ion bombardment. We carried out some early studies in this field with a variety of solids such as semiconductors, oxides and silicates [4]. As examples, gas release curves recorded with <sup>85</sup>Kr injected into ion bombardment disordered (amorphized) corundum and zircon are shown in Fig. 1. In general, the amorphous-crystalline transition occurs (and is accompanied by a distinct gas release stage) at a temperature between 0.3 and 0.6 times the absolute melting point of the solid. This gas release temperature is also related to the effective cation self-diffusion temperature and takes place at about 0.8 times this value. It was suggested that the amorphous-crystalline transition proceeds through the growth of the crystalline phase towards the bombarded surface so that the injected inert gas atoms are swept out as the disorder-crystal interface reaches the surface. Solids disordered by ion bombardment were investigated not only using ion injected inert gases but also using the classical emanation method by Balek [5]. The inert gas release technique was also used to study effects of neutron irradiation on Xe diffusion in various solids [6].

Annealing of the ion bombardment-produced amorphous phase can be achieved not only by thermal treatment of the sample but also by irradiating



Fig. 1. Release rate (dF/dt) of <sup>85</sup>Kr from zircon and corundum showing disorder annealing related peaks. <sup>85</sup>Kr was injected by ion bombardment at 10 keV.

it with intense light pulses from lasers. A study of <sup>85</sup>Kr release from implanted silicon specimens after delivering various amounts of energy from a neodymium glass laser was carried out by Sørensen [7]. This study demonstrated the wide range of applicability of ETA in this field. The ETA method seems to be particularly valuable for the determination of temperatures at which individual annealing stages take place. It does not give, however, direct evidence of the nature of the damage being annealed or on other characteristics of the disordered solid.

As to the future prospects and possible applications of ETA techniques in this field, the following suggestions may be of value.

From the methodological point of view, the technique of alpha-recoil implantation, in which  $^{222}$ Rn atoms are recoil injected into the sample from an external thin-layer  $^{226}$ Ra source under vacuum, could find useful applications here. In this way incorporation of radon atoms into a surface layer with an approximately exponential depth distribution of medium depth ca. 10  $\mu$ g cm<sup>-1</sup> could be obtained [8].

An interesting area of ETA application could be the study of disorder and disorder annealing created by low-energy cosmic particles (such as those present in solar wind) from materials in space. One could analyse in the laboratory specimens exposed to irradiation in space or one could consider injecting inert gas atoms into the specimens and measuring their release as a result of damage creation and annealing directly in the vacuum of the space environment.

Finally, studies of the thermal stability of amorphous metals are of increasing importance.

## SUMMARY

ETA can provide valuable information on the temperatures ranges in which individual disorder annealing stages (particularly the amorphouscrystalline transition) take place. Possible applications in studies of the effects of space radiation and of the stability of amorphous metals are suggested.

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