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Separation of radiation defects in Ni and Ni-C alloys under electron and neutron irradiation

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Abstract. Complex investigations of radiation damage of Ni and Ni-880 at. ppm C alloy under electron and neutron irradiation in the region of room temperature hardened and deformed state. In pure nickel, with the deformation microstructure, both in electron and in the neutron irradiation is observed separation of radiation-induced defects. When electron irradiation in the alloy Ni-C separation effect is observed, and when neutron irradiation there is no. This is due to the interaction of carbon atoms with radiation defects. The main sinks for radiation-induced defects are the areas with a high concentration of defects in cascades of atomic displacements.

1. Introduction
Operation of nuclear power plants of various types with great sharpness raises the problem of the radiation resistance of structural materials. At irradiation temperatures below 1/3 of the melting point the radiation damage is manifested in the accumulation of small clusters of radiation defects and the individual single vacancies. Nickel is a model material for the austenitic stainless steels. In nickel at room temperature vacancies are immobile and interstitial free to migrate.

Vacancies and vacancy clusters (VC) play an important role in radiation damage to metals and alloys, including radiation-induced structural phase transitions [1]. When cascadeless electron irradiation, VC formed by the free migration of vacancies. When cascade neutron irradiation vacancy clusters, besides formed directly in the displacement cascades. VC are efficient sinks of point defects and affect the radiation-induced diffusion processes [2,3]. In the austenitic stainless steels to reduce radiation damage used plastic deformation. In [4,5] theoretically studied the role of dislocation sinks on vacancy pore formation. Comparison of cascade neutron and cascadeless electron irradiation deformed and quenched metals and alloys reveals the role of the free migrated radiation defects and cascades of atomic displacements.

We was experimentally discovered in nickel the effect of the separation of radiation defects [6]. A substantial part of self-interstitials annihilated when migrating to the dislocation sinks and there lead a greater accumulation of vacancies in the deformed nickel. Carbon in solid solution (tens - hundreds of atomic ppm) can have a major effect on the properties of irradiated alloy as a result of its strong interaction with point defects [7,8]. The aim of this work is to identify the role of carbon atoms in the separation effect of radiation defects.
2. Materials and methods
For nickel doping carbon at 880 at. ppm by vacuum remelting, it is taken the nickel with additions of iron carbide. Solubility of carbon in the solid nickel decreases with decreasing temperature to 2400 at ppm at room temperature [9]. Nickel alloys were annealed at 1173 K in high vacuum for one hour followed by rapid cooling. Carbon in Ni-C alloys are in solid solution and thermally stable up to 850 K.

Electron microscopic studies were carried out on the microscope JEM 200CX. To delete a of vacancies created by deformation the sample was annealed at 450 K. The dislocation structure is completely preserved. Measurement of resistivity was conducted with standard four-probe measurement with error - 0.02%.

Irradiation of nickel and nickel alloys samples was performed of neutron dose (En> 0.1 MeV) F = (1, 10, 50)·10^{18} cm^{-2} and electron doses (Ee = 5 MeV) in the range (0-5)·10^{18} cm^{-2}. The temperature of the neutron irradiation is 300-320 K and 270-300 K of electron irradiation. For dose dependence on irradiation with fast neutrons  were used a set  of samples exposed to different doses. When electron irradiation was performed it is sequential irradiation with different doses of the same sample.

3. Experimental results
To investigate the microstructure were carried out electron microscopic studies. Recrystallized by slow cooling pure nickel has a dislocation density of at 10^{7}-10^{8} cm^{-2}. At the rapid cooling of pure nickel, a dislocation density is obtained of about 10^{9} cm^{-2} and at C-Ni alloys at 5·10^{9} cm^{-2}. At a plastic deformation it is formed cellular structure. The cell size decreases with increasing degree of deformation up to 90% from 700 nm to 200 nm. After low-temperature annealing for 0.5 hours at 450 K of deformed nickel the cellular structure is is stored. The volume of the cell have a density of dislocations 2,3·10^{10}cm^{-2} in the cell walls consisting of plexuses dislocation, the dislocation density is 4,8·10^{10}cm^{-2} and higher. At neutron irradiation it is observed vacancy clusters the size of 2-3 nm.

**Figure 1.** The dose dependence of resistivity growth for the pure nickel with varying degrees of deformation under electron irradiation at 270 K. Before irradiation, the samples were annealed at 450 K to remove deformation vacancies.

**Figure 2.** Dose dependence of resistivity growth for the alloy Ni-C with varying degrees of deformation under electron irradiation at 260 K.
The increase in resistivity when the nickel is deformed is the sum of the contributions from the dislocations and vacancies. These contributions may be divided. Changes in resistivity after annealing at 450 K, is determined by deformation vacancies. Using the value of the specific contribution of vacancies in pure nickel 0.23-0.28 nΩ·cm/at.ppm [7], we can estimate the amount of vacancies. At the degree of deformation of 40%, it is 67 at. ppm. The contribution given by the dislocations in deformed nickel corresponds to a change in the resistivity at recrystallization stage. At the specific contribution of dislocations in Ni, is equal to 9.4·10^{10} nΩ·cm^{-2} [10] the density of dislocations in nickel with a degree of deformation of 40% is approximately 5.6·10^{15} cm^{-2}; as a result of electron microscopy it is 9·10^{10} cm^{-2}.

Fig. 1 shows the dose dependence for pure nickel with different degrees of deformation. The greatest effect is observed when the separation degree of deformation of about 40%. This is in agreement with electron microscopic data on the dislocation density in the cells. Figure 2 shows the dose dependence of the alloy Ni-C with varying degrees of deformation. It can be seen that under electron irradiation of Ni-C alloy also observed the effect of separation. As the level of deformation up to 64% the effect is increased. Maximum effect, as in the case of pure nickel at 40% deformation was not observed.

To determine the effect of carbon on accumulation vacancies on Figure 3 shows the dose dependence for deformed and non-deformed alloy of pure Ni and Ni-C. The figure shows that the increase in the resistivity of the Ni-C alloy during irradiation less than pure nickel.

Figure 4 shows the results of neutron irradiation of Ni and Ni-C alloy in the deformed and non-deformed state. It is seen that the magnitude of increase resistivity under neutron irradiation is considerably higher than under the electron irradiation. On pure nickel effect of radiation defects separation is observed. At the Ni-C alloy separation effect is not observed. Increase resistivity in the deformed alloy is less than in the undeformed alloy.

From the change in resistivity under electron irradiation to a dose of 5·10^{18} cm^{-2} concentration of radiation vacancies is 30 at.ppm in undeformed nickel and 63 at.ppm in the deformed by 40% nickel. In the alloy Ni-C concentration of vacancies is 23 at.ppm in undeformed and 41 at.ppm in the
deformed by 48% nickel. At neutron irradiation it is generated vacancies and VC. In [11] it is shown that the contribution to the resistivity of vacancies does not depend on whether they are located individually or in clusters. Under neutron irradiation at a dose of 2·10¹⁹ cm⁻² the amount of vacancies will be in pure nickel in the recrystallized state 222 at.ppm, in a deformed state 307 at.ppm and in the alloy Ni-C - 327 at.ppm in the recrystallized state and 217 at.ppm in the deformed state.

4. Discussion

The efficiency of radiation defects sinks is determined by their capacity. Sinks capacity of dislocations in the volume of subgrain 1.5-2 times more than the sinks capacity of the walls subgrain [6]. Hence the initial sinks capacity in deformed nickel and Ni-C alloy is associated with the value of the initial residual resistivity \( \rho_0 \). During irradiation due to the accumulation of vacancies total capacity of sinks will only increase, and at higher doses go into saturation. The amount of accumulated during irradiation vacancies is determined by the resistivity growth \( \Delta \rho \). Thus it must be performed a linear relationship between the magnitude \( \Delta \rho \) and \( \rho_0 \). Due to the fact that the initial resistivity of pure nickel alloys and Ni-C differ, we used the value of the difference between the deformed and undeformed state for nickel and Ni-C alloy \( \rho_0(\text{def}) - \rho_0(\text{nodef}) = \Delta \rho_0 \). Figure 5 shows the results of such a comparison for nickel alloys and Ni-C irradiated by electrons to a dose of 5·10¹⁸ cm⁻².

![Figure 5](image.png)

**Figure 5.** The dependence of resistivity increase under electron irradiation (5·10¹⁸ cm⁻²) on the increase in the resistivity of plastically deformed nickel and Ni-C alloy.

As can be seen from the figure, there is a linear relationship. However, the separation effect of radiation defects in the alloy, Ni-C less than pure nickel.

At our irradiation temperatures the vacancies and the carbon atoms are immobile and mobile are just self-interstitials (SIA). Currently, is not enough studies of the interaction of atoms and point defects in nickel. As the data modeling [12], the carbon atoms in nickel can be effectively trapped at the SIA and vacancies. In [7] it is assumed that the complex SIA - atom C annealed at 70 K. Thus we can assume that in this case, under irradiation it is move are not only SIA but also the complexes SIA - atom C. In its migration such complexes can interact with other atoms C to form carbon clusters. Thus, the formation of carbon clusters are carbon output from the solid solution and decrease resistivity. The contribution to the resistivity of carbon - 3.5·µΩ cm/at.% [13].

The number of generated displacements per atom in the alloy and pure nickel at any dose of irradiation is the same. At neutron irradiation in pure nickel separation effect is observed, as at the electron irradiation. In the alloy Ni-C situation is reversed - the increase in the resistivity of the deformed alloy less than undeformed about half. The values of the resistivity increments under neutron irradiation in Ni-C alloys greater than in the pure nickel. When migrating complexes CMA-C atom it can be formed carbon clusters, as under the electron irradiation. When migrating complexes
SMA-C atom in the areas of cascades of atomic displacements it is a high probability clustering, containing in the structure a few atoms and CMA and stable at irradiation temperature. This leads to increased accumulation of radiation defects and therefore to the large increase in resistivity in Ni-C alloys compared to pure nickel. In this case, a large concentration of dislocation sinks in deformed Ni-C samples compared to undeformed increases annihilation migrating complexes atom C-SMA at dislocations and reduce the growth of resistivity.

5. Conclusion
Complex investigations of radiation damage of Ni and Ni-alloys 880 atomic ppm C under electron and neutron irradiation in the region of room temperature quenched and deformed state. According to the research it may be concluded:

1. In pure nickel, with the deformation microstructure, both under electron and in neutron irradiation in the vicinity of room temperature is observed the separation of radiation defects. Under electron irradiation separation of radiation defects have the maximum at degree of deformation of about 40% and are determined by the dislocation microstructure and its spatial distribution.

2. When the alloy Ni-C irradiated by electrons it is also observed the effect of the separation of radiation defects. The growth of resistivity is constrained by the presence of carbon atoms through the formation of migrating complexes C atom - own interstitials, leading to the removal of carbon from the solid solution.

3. Upon irradiation Ni-C alloys with neutrons, the effect of radiation defects separation are not observed. This is due to the effect of carbon on the formation of stable clusters consisting of carbon atoms and self-interstitials in the areas of displacement cascades.

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