Phase diffusionless $\gamma \leftrightarrow \alpha$ transformations and their effect on physical, mechanical and corrosion properties of austenitic stainless steels irradiated with neutrons and charged particles

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Abstract. The work presents relationships of $\gamma \rightarrow \alpha'$ and $\alpha' \rightarrow \gamma$-transformations in reactor 12Cr18Ni10Ti and 08Cr16Ni11Mo3 austenitic stainless steels induced by cold work, irradiation and/or temperature. Energy and mechanical parameters of nucleation and development of deformation-induced martensitic $\alpha'$-phase in the non-irradiated and irradiated steels are given. The mechanisms of localized static deformation were investigated and its effect on martensitic $\gamma \rightarrow \alpha'$ transformation is determined. It has been shown that irradiation of 12Cr18Ni10Ti steel with heavy Kr ions ($1.56\text{MeV/nucleon}$, fluence of $1 \times 10^{15} \text{cm}^{-2}$) results in formation of $\alpha'$-martensite in near-surface layer of the sample. Results of systematic research on reversed $\alpha' \rightarrow \gamma$-transformation in austenitic metastable stainless steels irradiated with slow (VVR-K) and fast (BN-350) neutrons are presented. The effect of annealing on strength and magnetic characteristics was determined. It was found that at the temperature of $400^\circ\text{C}$ in the irradiated with neutrons samples (59 dpa) an increase of ferromagnetic $\alpha'$-phase and microhardness was observed. The obtained results could be used during assessment of operational characteristics of highly irradiated austenitic steels during transportation and storage of Fuel Assemblies for fast nuclear reactors.

1. Introduction
In recent years, researchers, working in the area of radiation materials science, are interested in effects of temperature, deformation and irradiation on phase $\gamma \leftrightarrow \alpha$ transformations taking place in austenitic stainless steels. According to [1] this interest is related to the fact that after a long term operation of WWR, diffusionless transformations (a second order effects) start to play an important role in developing corrosion and mechanical properties of reactor materials. This research studies diffusionless martensitic transformations in irradiated structural materials of nuclear reactors. It is known that high-alloyed austenitic stainless steels and alloys in accordance with thermodynamics laws (figure 1) and phase diagrams (figure 2) in the combined effects of stress, strain, radiation and temperature exhibit the metastability, showing the whole number of phase-structural transformations, radically affecting operational characteristics [2-5]. In figure 1 $T_0$ is the equilibrium temperature of prior austenitic ($\gamma$) and martensitic ($\alpha'$) phases, $\Delta \sigma_m^{\gamma \rightarrow \alpha'}$ is the critical value of the chemical driving force required to initiate $\gamma \rightarrow \alpha'$ - transformation under cooling with no load. Martensitic transformation
may begin at $T_1$, $M_S < T_1 < T_0$ if the external load eases the critical nucleus formation, i.e. if it decreases at some value $U$ required chemical driving force $\Delta \sigma_{M,S}^{\gamma\rightarrow\alpha}$.

![Figure 1. The free energy changing as a function of temperature [3].](image1.png)

2. The role of stacking fault energy (SFE) in $\gamma\rightarrow\alpha'$ transformation

One of the important characteristic that causes structural-phase transformations in austenitic steels is stacking fault energy ($\gamma_{sfe}$) of metal material, which largely depends on concentrations in the austenitic solution such element as C, N and Ni [6]. In general it is considered that in stainless steels SFE increases with Ni and C; whereas Cr, Mn and Si and N decrease SFE. The attempts to establish simple linear correlations between SFE and element concentrations in the stainless steels lead, in particular, to the following expressions:

$$\gamma = -53 + 6.2\%Ni + 0.7\%Cr + 3.2\%Mn + 9.3Mo \ [MJ/m^2] \ [7];$$
$$\gamma = 1.2 + 1.4\%Ni + 0.6\%Cr + 17.7\%Mn - 44.7Si \ [MJ/m^2] \ [8];$$
$$\gamma = 16.7 + 2.1\%Ni - 0.9\%Cr + 26\%C \ [MJ/m^2] \ [9].$$

Martensitic $\gamma\rightarrow\alpha'$ - transformation results in the transition from the initial austenitic FCC lattice ($\gamma$ - solid solution) to martensitic BCC lattice ($\alpha'$ - phase), what occurs diffusionless with the saving austenitic initial elementary composition in accordance with orientation ration of Kyrdymov Zaks $(111)_\gamma \parallel (011)_{\alpha'}, [\bar{T}01], [\bar{T}\bar{T}1]_{\alpha}$ (figure 2, a). More details on martensitic transition starting temperature, the value of the applied stress ($\sigma$) and temperature dependence of the stable austenite yield strength ($\sigma_y$) are illustrated in figure 2, b. At the temperature $M_S$ first martensite crystals arise without any external load. At temperature range $M_S<T<M'_f$ the martensite crystals formation is initiated by elastic stresses $\sigma<\sigma_y$.

![Figure 2. Phase $\gamma\rightarrow\alpha'$-transition under the effect of stresses and temperature [4].](image2.png)
The obtained under these conditions $\alpha$-martensite was named as the stress-induced martensite. At $M'_s < T < M_d$ plastic deformation precedes the martensite appearance, which was named as deformation-induced martensite.

During cold deformation austenitic steels are characterized by low stacking fault energy; $\alpha'$-martensite arises at the intersection of the split dislocations and the phase transformation takes place at temperatures $M_s - M_d$ (figure 3).

![Figure 3. The incipience of martensitic $\alpha'$-phase during deformation at the intersection of stacking faults.](image)

Above the $M_d$ temperature is not possible to induce the martensitic transformation by plastic deformation. Each stainless steel is characterized by $M_d$ temperature setting value. The work [10] suggested the empirical temperature relationship $M_d$ from composition of alloying elements for chromium-nickel steels:

$$ M_d(\degree C) = 551 - 462(C+N) - 9.2Si - 8.1Mn - 13.7Cr - 29(Ni+Cu) - 18.5Mo - 68Nb \quad (2) $$

We have found that for 12Cr18Ni10Ti steel the $M_d$ temperature above which deformation-induced $\alpha'$-phase is not formed is 100$\degree$C both for unirradiated and irradiated with neutrons samples up to the fluence of $2 \times 10^{20}$ n/cm$^2$. Using the energy approach to the consideration of the $\alpha'$-phase formation and destruction of metastable reactor steels the study proposed a new definition of $M_d$ as the temperature at which the energy required for the starting $\gamma \rightarrow \alpha'$ transition becomes equal of mechanical energy which one needs to impart to the material to its failure. Thus at deformation temperatures more than $M_d$ the sample fails before it has time to perform $\gamma \rightarrow \alpha'$-transition (figure 4).

**3. Critical parameters of deformation-induced $\alpha'$-phase formation**

In addition, it was found [11] that martensitic $\alpha'$-phase in the irradiated and deformed metastable steels is characterized by a low stacking fault energy values, and it forms only when the cellular dislocations start to dominate at a certain stage of the defect structure evolution. At the intersection of
the cell walls, consisting of a number of split dislocations with $\varepsilon$-phase (hcp), increases the probability of $\alpha$-phase formation (figure 5) at a reaction of $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ transformations.

![Figure 4](image)

**Figure 4.** Temperature dependences of mechanical work, needed to originate martensitic $\gamma \rightarrow \alpha'$-transformation and to destruct unirradiated 12Cr18Ni10Ti steel samples at deformation.

![Figure 5](image)

**Figure 5.** The evolution of defect structures and deformation-induced $\gamma \rightarrow \alpha'$-phase transformation in in metastable stainless steels at low temperatures.

Generally, at low temperature static tension $\alpha'$-martensite of deformation forms at some critical deformation values $\delta_{cr}$, stresses $\sigma_{cr}$ or mechanical work density $A_{cr}^{\gamma \rightarrow \alpha'}$, and with increasing doses of neutron irradiation values $\delta_{cr}$ and $A_{cr}^{\gamma \rightarrow \alpha'}$ decrease and $\sigma_{cr}$ remain almost unchanged what is shown in the experimental results (figure 6 and table 1).
Figure 6. The formation and development of the deformation-induced martensitic $\alpha'$-phase in 12Cr18Ni10Ti steel samples, non-irradiated and irradiated with slow neutrons (VVR-K).

Table 1. Critical nucleation $\alpha'$-phase parameters at stretching non-irradiated and irradiated with neutrons 12Cr18Ni10Ti steel samples ($T_{\text{test}} = 20^\circ C$, $v = 0.1 \text{ mm/min}$).

| Sample number | Fluence (n/cm$^2$) | $\sigma_{cr}$ (true) (kg/mm$^2$) | $\delta_{cr}$ (%) | $A_{cr-\alpha'}$ (MJ/m$^3$) |
|---------------|-------------------|-------------------------------|------------------|------------------|
| 510           | 0                 | 62                           | 20.8             | 60               |
| 356           | $2 \times 10^{18}$ | 62-63                        | 10.2             | 55               |
| 369           | $5 \times 10^{18}$ | 59                           | 9.1              | 40               |
| 95            | $2 \times 10^{20}$ | 61-65                        | 4                | 30               |

4. Kinetics of martensitic $\alpha'$-phase accumulation at deformation

Being formed, $\alpha'$-phase continues to accumulate in the deformed sample and as it turned out, in all of randomly selected for observation sample areas (except neck), $M_f$ accumulation occurs equally at the same power law. To describe the kinetics of deformation-induced $\gamma \rightarrow \alpha'$-transformation in the irradiated reactor steel the equation of Ludvigson-Berger [12] was chosen.

$$\frac{M_f}{1 - M_f} = Ae^B,$$

where the A parameter characterizes a tendency of deformed steel to martensitic transformation; B is the autocatalysis index. As a result, it was found that the experimentally determined B coefficient (B≈3.7) is similar for 12Cr18Ni10Ti steel samples both non-irradiated and irradiated with neutrons ($2 \times 10^{20}$ n/cm$^2$) [13, 14]. This is probably due to the fact that the neutron irradiation affects mainly positively on $\alpha$-phase nucleation and facilitates it by creating defect areas. At the same time, originated, martensitic lens hardly develops in hardened by defects irradiated austenitic matrix that eventually leads to the formation of a large number of small plates of $\alpha'$-phase.

5. $\gamma \rightarrow \alpha'$-transformation and localization of plastic deformation

It was shown that martensitic $\gamma \rightarrow \alpha'$-transformation plays a big role in localization processes of plastic deformation. The resulting $\alpha'$-phase is stronger than the austenitic one and this induces localized inhomogeneous deformation in the form of strips, consistently and quickly moving along the working length of the sample (figure 7), and then transformed into the neck.
Figure 7. Strips of localized deformation in the 12Cr18Ni10Ti steel irradiated with neutrons.

It was also established that at the pre-destruction stage the maximum number of $\alpha'$-ferrophase contained in that section of the working part of the sample, in which the destruction will happen. Thus, the precipitation kinetics of $\alpha'$-phase for different cross-sections of the sample may help to monitor the process of localization and judge the speed of shape changes. The fact of the percolation possibility of $\gamma \rightarrow \alpha'$-transformations in metastable austenitic stainless steel during deformation can be used to detect the early signs and origin place of necking in a deformed sample.

Figure 8 shows that, given the localization of deformation kinetics of accumulation $\alpha'$-martensite in the neck has an increasing character, predicted by various theories. Here the value of the parameter $B$ for neutron-irradiated steel less than the non-irradiated. Thus, deformation-induced martensitic $\gamma \rightarrow \alpha'$-transformation can have a significant effect on the local deformation in the reactor stainless steels. Estimates of the strips moving rates in unirradiated ($V_{unirr}$) and irradiated ($V_{irr}$) with neutrons ($2 \times 10^{20}$ n/cm$^2$) 12Cr18Ni10Ti steel samples deformed at 20$^\circ$C at a strain rate of 0.5 mm/min, showed that at the deformations $\varepsilon_{unirr} = 33\%$ and $\varepsilon_{irr}=18\%$ the speed of strips in the irradiated sample is higher than in unirradiated sample and constitute $V_{unirr} = 6.5$ mm/min $V_{irr} = 12.5$ mm/min.

6. Deformation-induced wave
In some cases, such as when deformation (T = 20$^\circ$C) highly irradiated (56 dpa) 12Cr18Ni10Ti steel sample, we observed the movement modeled on macrowaved deformation that led to the achievement of anomalously high plasticity brittle material after irradiation [15-18].

At the same time initial round markers, applying the paint to the surface of the sample, have evolved into ellipses, and at the diagram of stretching fixed plateau with the same value of the true flow stress (figure 9). It is found that in this case the rate of “wave” was $\sim 0.04$ mm/s at a grip rate of 0.008 mm/s.
Figure 8. Kinetics of the formation and accumulation of deformation-induced $\alpha'$-phase in the 12Cr18Ni10Ti steel: a) Engineering stress-strain diagram (4), kinetic curves of $\alpha'$-phase (1) in localization zone (Mf) and (2, 3) - near the neck of irradiated steel up to $5 \times 10^{22} \text{n/m}^2$ (at inset martensitic phase distribution along the sample); b) the trend $\alpha'$-phase curve in neck (inset TEM image and electron diffraction pattern $\alpha'$-martensitic phase).

Figure 9. Deformation wave (a), its circuit (b) and the engineering stress-strain diagram of high irradiated samples of chromium-nickel steels (c) for cases of "no wave" (1.3) and "with a wave" (2).

The conducted systematic investigations of localized deformation found that a greater role of martensitic $\gamma \rightarrow \alpha'$-transformation plays during the formation and development of a stable neck in a deformed sample of metastable austenitic steel [19-21].

By analyzing the results of numerous experiments carried out to investigate the effect of neutron irradiation dose on the mechanical characteristics and power characteristics of reactor of austenitic steels, it was found that irradiation to some critical neutron fluence $F_t=3 \cdot 10^{22} \text{n/cm}^2$, $\gamma \rightarrow \alpha'$-transformation in steels containing radiation defects may start spontaneously, without any additionally induced cold deformation (figure 10).
Figure 10. The effect of neutron fluence on the critical deformation (left) and energy density (right) required for $\gamma \rightarrow \alpha'$-transformation in 12Cr18Ni10Ti steel ($E>0.1$ MeV).

7. Irradiation-induced martensite

In the past we have repeatedly registered that the austenitic stainless steel samples were cut from different levels of the wall covers of spent fuel assemblies of the BN-350 nuclear reactor, detected the presence of $\alpha'$-phase (figure 11).

Figure 11. Formation $\alpha'$-austenitic steels under irradiation with fast neutrons (12Cr18Ni10Ti steel, BN-350, CC-19, «0mm», 56 dpa, 370°C) (a); Frank loops, x50000 (b).

It was suggested that formation of martensitic phase is related to Frank loops. According to TEM studies these were the main radiation defects in irradiated material. This result, in particular, is confirmed by data obtained at ORNL (USA) under neutron irradiation of 304 steel (up to 12 dpa) with different silicon content in BOR-60 [24].

Based on the analysis of numerous experimental data it has been suggested that in 12Cr18Ni10Ti austenitic stainless steel ferromagnetic martensitic $\alpha$-phase can be formed under neutron irradiation
without introducing additional deformation. Apparently, in this effect a big role is assigned to the stored energy; upon reaching a critical value in the material $\gamma \rightarrow \alpha'$-transformation takes place. Using this approach $\alpha$-phase in the austenite matrix should be formed not only in the case of neutron irradiation, but also by irradiation with charged particles. Indeed, as has been shown in [25] using EBSD method, that irradiation of 12Cr18Ni10Ti steel with heavy krypton ions with an energy of 1.56 MeV/nucleon at a fluence of $10^{16}$ particles/cm$^2$ leads to formation of $\alpha$-phase (figure 12). It was suggested to call this phase as irradiation-induced martensite by analogy with established concepts in the literature: "martensite of deformation", "stress" and "heat treatment" [26].

**Figure 12.** $\alpha$-martensite irradiation in 12Cr18Ni10Ti steel after irradiation with heavy ions in the DC-60 accelerator (Kr 1.56 MeV/ nucleon) $\sim 10^{16}$ particles/cm$^2$. Quantity $\alpha$-martensite (BCC-lattice) $\sim 9\%$; $\varepsilon$-martensite (HCP-lattice) $\sim 2\%$.

It is worth noting that the formation of $\alpha$-phase in the US austenitic steels was studied earlier in [27-30], and in the Russian steels this effect was discovered and described in [31, 32].

Taking into account the currently available information, we concluded that a phase transition $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ can occur during irradiation when the defect structure evolves directly to the Frank loops at the by mechanisms proposed in [33]. The mechanisms impose restrictive conditions on the size of loops or, in other words, the stacking fault energy of steel. This idea found even greater confirmation when anomalously large stacking fault tetrahedrons were observed in the structure of steel samples irradiated with fast neutrons (figure 13). Both the stacking faults and Frank loops are related to the appearance of ferromagnetism. In fact, taking into account the generally accepted mechanism of formation of the tetrahedron, at every plane of which develops a defective dislocation loop and on the ribs can occur intersection of loops, i.e, $\varepsilon \rightarrow \alpha'$ transformation.

**Figure 13.** Tetrahedrons of stacking faults and $\alpha$-phase in the microstructure of 08Cr16Ni11Mo3 austenitic stainless steel, irradiated with fast neutrons (BN-350, H-214(II), $\approx 0$ mm, 15.6 dpa $T_{irr}$=337$^\circ$C, 9-10$^{22}$ n/cm$^2$).

It has been suggested [26] the following formation scheme of $\alpha$-phase in the metastable austenitic steel irradiated with neutrons at various temperatures to various damaging doses (figure 14). Irradiation-induced martensite is formed in the region 1 and is characterized by relatively low values of damaging dose (10-60 dpa) and irradiation temperature (200-400$^\circ$C). In region 3 is also formed $\alpha$-
phase, but its nature is different – it is likely to be deformation (stress)-induced martensite. As the structure of \( \alpha \)-phase is the bcc lattice, which itself is loose and therefore swells less than the fcc structure, so in the two temperature-dose areas 1 and 3, we can expect that swelling therein will be relatively less. Some experimental confirmation of this idea is obtained in [34].

8. Reversed \( \alpha' \rightarrow \gamma \)-transformation

Stated above is attributed to \( \alpha \)-martensite induced by deformation or irradiation of austenitic reactor steels. Being formed, \( \alpha \)-phase can exist in the crystal as long as the material temperature is not increased to \( T = 350-400^\circ C \). At higher temperatures the amount of \( \alpha' \)-phase will continuously decrease as temperature increases and upon reaching \( T = 700-800^\circ C \) the microstructure will be completely annealed (figure 15a) with the formation of precipitation-hardened austenite [35]. Thus process of \( \alpha' \rightarrow \gamma \)-transformation is a multistage process. During magnetization occur 3 or 4 maximums (peaks), which position on the temperature scale depends on the radiation dose [36].

Analysis of the experimental data including results of Mössbauer, X-ray diffraction and TEM studies allows to assume the following sequence of changes in the structure, elemental and phase composition occurring during annealing of cold stainless steels in the identified stages. At \( 400^\circ C \) the mobility of the chromium atoms increases leading to separation of the solid \( \gamma \)-solution with the formation of concentration inhomogeneities - spherical Guinier-Preston zones enriched with chromium. In the temperature range of 400-450\(^\circ C \) \( \alpha' \)-phase and Frank loops start to anneal and the swelling rate reaches 1\% / dpa. It is worth noting that in some cases during the annealing of cold or irradiated steel samples around \( 400^\circ C \) it was observed an increase of \( \alpha' \)-phase and this effect was also accompanied by an increase in microhardness and thermal effects (figure 16) [37]. In the range of 400-475\(^\circ C \) occurs annealing of the newly formed \( \alpha' \)-phase having a microscopic size. This annealing step is athermal. In the temperature range of 450-675\(^\circ C \) an opposite \( \alpha' \rightarrow \gamma \)-transition occurs, which is carried out by a cooperative rearrangement of atoms by martensitic mechanism. This process is accompanied by a decrease in volume of the material. The \( \alpha' \rightarrow \gamma \)-transition ends by formation of netted dislocation structure in austenite.
Figure 15. a) The inverted $\alpha' \to \gamma$ martensitic transition in the stainless steels, irradiated with neutrons (BN-350); b) $400^\circ$C – The starting temperature of inverted $\alpha' \to \gamma$- transformation, annealing of Frank loops and swelling at a rate of 1%/dpa.

Figure 16. a) The observed effect of an increase in $\alpha'$-phase during annealing of deformed 12Cr18Ni10Ti steel samples irradiated with neutrons to a damaging dose of 55.7 dpa; (in the insert – thermal effects accompanying $\alpha'$-martensitic transition (200-400°C – heat absorption, 400-600°C – heat generation)); b, c) 08Cr16Ni11Mo3 steel microstructure (BN-350, FA H-214(II), «<1200mm», 0,25 dpa; b) before annealing; c) after annealing at 800°C (1 hour).

A decrease of annealing rate in the area of 550-700°C is associated with an increase in the volume fraction of $\alpha'$-phase due to the fact that heating at temperatures 400-700°C is caused by ageing of steel, the formation of carbide and intermetallic phases and resulted in austenite destabilization and the formation of secondary $\alpha'$-martensite due to depletion of solid solution in austenite – stable elements – C, N and Ni. In the range of 550-800°C magnetization of steel is decreasing due to the annealing of secondary $\alpha'$- martensite. This third step of the martensitic transformation to austenite occurs by a mechanism controlled by diffusion.
9. The effect of $\alpha'$-martensitic phase on corrosion in 12Cr18Ni10Ti stainless steel

The performed studies of the deformed and irradiated austenitic stainless steels have shown a decrease in the resistance of structural materials of nuclear reactors on fast neutrons to localized corrosion due to the formation and growth of induced deformation and irradiation $\alpha$-martensite [37]. It was determined that samples of 12Cr18Ni10Ti steel irradiated with neutrons ($10^{19}$ n/cm$^2$) in the VVR-K reactor and deformed at $-60^\circ$C to 30% strain or more, apart from structural inhomogeneity of the phase are characterized by phase heterogeneity and the emergence of major corrosion pits, concentrated near the deformation strips and grain boundaries, where martensitic $\alpha'$-phase is mainly accumulated.

In [38] gives data on corrosion resistance exposed to high damaging doses (~59 dpa) 12Cr18Ni10Ti steel samples cut from the walls of the covers of spent fuel assemblies from the operational life of the reactor BN-350. It was found that samples cut from different levels of from the center of the reactor core, contain different amounts of magnetic $\alpha'$-phase, what correlated with the features of the defect structure (presence Frank loops and stacking fault tetrahedra). Established the intergranular nature of corrosion damage to the inner wall surface of the cover screen FA H-214 (1) at the level of "75 mm" from the center of the CAZ, where radiation damages are particularly noticeable. Figure 17 shows the deformation dependence of mass loss of steel samples 12Cr18Ni10Ti in aggressive solution (10% FeCl$_3$, 500 hours), which shows ambiguous changes in corrosion damage on the degree of cold deformation.

![Figure 17](image)

**Figure 17.** Effect of deformation martensite at resistance to pitting corrosion of cold deformed 12Cr18Ni10Ti steel (given in parentheses the volume content $\alpha'$-martensitic phase in %).

10. Conclusions

There was carried out a complex research on materials science of structural materials for fast reactors and thermonuclear installations, and presented data analysis results. It is shown that in complex-alloyed austenitic stainless steel an intensive external influences (radiation, deformation, temperature) may result in phase-structural transformations, including direct ($\gamma \rightarrow \alpha'$) and reversed ($\alpha' \rightarrow \gamma$) martensitic transformations, largely determining the operating characteristics of these materials. Considered some features of formation and development of deformation (stress)-induced and irradiation-induced martensite. Established the influence of $\gamma \rightarrow \alpha'$-transformation at localized deformation in the form of strips and stable necking. Obtained data allow to suggest that $\alpha'$-phase,
formed in the austenitic stainless steel, has a significant impact on corrosion properties and play an important role in long-term (>50 years) storage of spent fuel assemblies of fast reactors.

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