

# Study of Radiation Damage Characterization in Metals

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**Abstract** - Nuclear energy emerged as reliable source of electricity. Its economic life must be successfully predicted to continue to make further advanced power plant having extended work life. Study of radiation damage to the structural components will lead to further development of advanced materials. Also discussion on five key bulk radiation degradation effects (high temperature helium embrittlement; radiation-induced and modified solute segregation and phase stability; irradiation creep; void swelling; and low temperature radiation hardening and embrittlement) and a multitude of corrosion and stress corrosion cracking effects (including irradiation-assisted phenomena) that can have a major impact on the performance of structural materials in nuclear reactor.

**Key Words:** Embrittlement, SEM, TEM, Irradiation creep, Void swelling, Radiation hardening.

## 1. INTRODUCTION

In nuclear power plants design of components is extremely complex and requires the expertise of a range of disciplines. The reactor core is the responsibility of nuclear engineers and must be designed with numerous constraints on how the reactor is operated. These constraints include performance of system in terms of reliability and specific safety with economic performance. [1][2]

Nuclear reactors have very harsh environment for components and also cumulative effects of high temperature water, stress, vibration, and in the last major effect of an intense neutron field. Performance is reduced because of degradation also the fear of sudden failure with materials aging and cable/piping degradation. Over 20 different metal alloys are employed within the primary and secondary systems light water reactor. The dominant forms of damage may vary between different reactor systems and subsystems.

However, material damage of any component can have an important role in the safe and efficient operation of a nuclear power plant. Periodic checks and parts replacement can reduce these factors; still there is chance of failure. For extended reactor work life, many components must bear the reactor environment at temperatures near or slightly higher than their original design limit for very long times. This may lead to failure for some components and may introduce new damage modes. While many components in reactor (except the reactor pressure vessel and concrete structures) can be replaced, it may not be advisable as the cost parameter is concern. Therefore, understanding, controlling, and

mitigating materials degradation processes are key priorities for reactor operation and life extension. The Pressure Vessel is the major equipment of a water nuclear reactor. It works as shield against radiation spread and material with suitable fracture toughness should be used either normal operation and maintenance cycles or under accidents. Neutron irradiation can degrade the fracture toughness of these steels. To increase life of nuclear reactor through inspection of this irradiation induced damage is required.

The most critical components whose reliable operation is needed is structural components within the reactor core (stainless steel) failing which may have severe consequences. There are a number of issues that must be considered and evaluated when considering extended reactor life-times, including thermal aging, fatigue, corrosion, and irradiation damage. The extended exposure of internal systems to elevated temperature induces a variety of phase transformations and potentially reduces fracture toughness. Fatigue can be concern for existing service conditions for a number of different components. Other major key factors addition to high temperatures is intense neutron fields. Components should also sustain corrosive environment in nuclear reactor. Stress in component may lead to stress-corrosion cracking (e.g. stress corrosion cracking, stress corrosion cracking due to irradiation). Since the corrosion mechanism generally varies with location in the reactor, A various other different mechanisms may also be operating at the same time in that environment. As far as safe and reliable operation of reactor is concern moderate levels of corrosion is avoided Because of this the different corrosion mechanisms should be studied and analysed for various reactor components. Finally, since the materials are used in an intense high-energy neutron field, there should be fine microstructural stability because of neutron damage. In modern Reactor there are three physical Barriers between Reactor and radioactive fuel. Concrete wall containment, pressure vessel and last is fuel cladding of which the pressure vessel and fuel cladding are made of metals and being exposed to neutrons. Aim of this literature survey is the characterization of neutron irradiation damage in structural materials. In the past we have come across various major commercial nuclear site accidents mainly related to release of radioactive materials in the environment consequences loss to human life. So study of radiation damage is itself is of a prime importance for safe and efficient operation nuclear power plant.

## 2. Types of radiation damage

In nuclear power plant various materials are used, in that metals will be our main focus to study. Because the main structures like pressure vessel, Fuel cladding are made of metals.

Nuclear reactor works at high temperature during fission reactions with bombardments of neutron having fast and intermediate spectrum on components of reactor. The 'fast' neutrons (0.1-10MeV) are a main damage to nuclear reactor components. The damage mechanism is the displacement of atoms from their lattice positions as a result of elastic collisions with neutrons. The displacement occurs when the transferred energy in the collision must exceed the displacement energy threshold value. Particularly the value for metal is 20-40 eV for metals [3].

When going through the crystal lattice, the bombarding species interact with the lattice atoms and lose some of their energy to these atoms. These processes can be accompanied by damage to the crystal lattice, in general of three types production of lattice atoms shifted out of their regular lattice positions, displacement damage, Changes in the chemical composition by stopping of the bombarding particles (called ion implantation) or capture of particles in the atomic nucleus with consequent transmutation and last is excitation of electrons and ionization of atoms (which does not produce permanent damage in metals).

These mechanisms vary according to different temperature and damage levels. Radiation Hardening is predominant in BCC metals at low temperatures which also results in loss of fracture toughness. This is due to dislocation motion being hindered by the radiation induced defect clusters. This phenomenon occurs at particularly low doses (0.001 to 0.1 dpa) and thus this defines the operating temperature limit at lower scale for the structural materials which are being exposed to neutrons. [6]

There are five main mechanisms for neutrons in the 0.1 to 10MeV range, by which the damage to structural materials occur within nuclear reactor. Includes helium embrittlement because of high temperature, creep due to Irradiation, volumetric swelling from void formation, radiation hardening embrittlement and precipitation caused by phase instabilities. All these mechanisms are vary according to different temperature and damage levels.

Strengthening of metals can be possible with Irradiation and these mechanisms are named as: source hardening and friction hardening. In source hardening dislocation is multiplied and unlocked because of stress caused by plastic deformation. Introduction of defect clusters in metal forms Frank-reed dislocation source by pinning or locking. So before this phenomenon expand, multiply and operate with influence of stress unpinning of dislocations must be done from defect.

## 3. Radiation damage characterization

As we have studied the various radiation damage mechanisms and issues facing specific materials being used. To be proposed for use in reactor design. There are many methods available to scientifically test and study these materials to determine their suitability for use in reactors.

To analyse the damage to which material is subjected, in reactor or in any radiation environment we must look at ways to perform or simulate the neutron damage. We will discuss the methods as found in the literature for performing or simulating the radiation damage in materials, we will then discuss the most common methods used to characterize this damage.

Two methods generally used to perform radiation damage on a material specimen are neutron irradiation and ion irradiation. In neutron irradiation we place material in a nuclear reactor for a given period of time until the dose required for the experiment has been obtained, Temperature control in this case may also be possible. The main problem with neutron irradiation experiment is slowness. Irradiation rates are very slow and the required dose may take years to achieve, also extended time used for this will make it expensive to perform. To overcome this problem activation of material should be done to deal with neutron irradiated samples. Once a sample is removed from the reactor, it may possible that the material itself may be radioactive due to transmutation reactions that occur when the neutrons collide with certain elements within the material. For this activation of material special handling is required, typically hot cells and special instrumentation is required to examine the radioactive materials following safe concerns. [8]

Other method of charged particle or ion beam irradiation is widely used over several years to understand radiation damage, with earliest works by kinchin and Pease in mid 1950s. Ion beam irradiations allow for more rapid and approximately 10 to 1000 times faster than that of neutrons because of lesser time it also results low cost, but high temperature makes difficult to interpret the data other benefit of this method is that dose, dose rate, temperature, chemical and mechanical environment these parameters can be controlled. In most of literature comparison of pros and cons of these two methods is discussed. The main limitation for charged particle irradiation are sample sizes, sample sizes are should be kept of small sizes which causes disproportionate surface effects, very large irradiation rates can affect rate dependent reaction pathways when replicating lower damage rates in a neutron irradiation more cumbersome to interpret. [9]

Energy of the particle in two systems is different as there is fundamentally large difference in the way ions and neutrons are produced. Charged particle has same energy as they are produced in an accelerator but not with neutrons. Neutrons which are produced in reactor has an energy spread over

several orders of magnitude depending on position where sample placed in reactor vessel, Energy of the neutrons will vary between different reactors ITER-International thermonuclear experimental reactor, FFTF-fast flux test facility, HFIR-High flux isotope reactor PWR- Pressurized water reactor [10]

Moreover there is one more factor which varies greatly between two system is depth of penetration. charged particles loses energy at rapid rate as there is loss in electronic energy when particle interacts with the metallic substrate .on the counterpart Neutrons being electrically neutral can penetrate up to much higher depth. The dose rate variation between charged particles and neutrons is considerable because the ion beams produces lager dose rates compared to neutrons, Kulcinski et al. [11] shown that charged particles will cause over 100 times more damage at the sample sur- face than neutrons as compared to the neutrons caused.

As discussed above transmutation reactions that occur with neutron irradiations, ion irradiation causes minor to zero residual activity allowing us to examine the samples as soon as the irradiation performed. And we should also consider how the charged particle irradiation might be able to simulate these transmutation reactions if we understand any material in a neutron environment.

Transmutation reactions in reactor also cause the production of helium and hydrogen as a result of fast neutron collisions with the nucleus of certain elements within elements. Most specific is the radiation damage by the production of helium by transmutation of nickel and boron. Helium and hydrogen arising from transmutation reactions will interact in ways that cannot be reproduced experimentally using charged particle irradiations, makes it another limitation of this technique as compared to neutron. Hydrogen formation is more common because electrolysis of water in a fission reactor, not because of transmutation. Multi beam irradiations, is not a complete true representation of neutron irradiations. Kumar [12] proved that dual beam irradiations undertaken a fusion experiment contained artefacts which were not representative in a neutron fusion environment citing interactions with self-ion generated interstitials and helium as the artefact.

With simultaneous ion irradiation important synergies may develop which depends upon dose leading to further complication in experimental interpretation. The use of simultaneous multiple beam ion accelerators is the most common method as it allows the implantation of both helium and hydrogen ions at the same time and or a heavy ion beam may also be employed for ballistic damage.

As we have discussed methods for performing radiation damage experiments with neutrons or charged particles, now we will see methods to characterize this radiation damage as found in the literature.

As per discussion above there are many major factors that influence the selection of materials for intended exposure to radiation damage. That Includes ductility, stability, mechanical strength, susceptibility and neutron absorbing characteristics to induced radioactivity. These properties which are mostly determined by mechanical testing set a benchmark for the material's ultimate suitability for use in a reactor. When ion implantation or neutron irradiation has been tested on a material it is these properties that can be examined to evaluate the materials performance; however research is more focused on the fundamental radiation damage mechanisms which occur before and after radiation damage. In the literature electron microscopy has been found to be a very widely used method for material characterization in radiation damage studies. Coupled with some other techniques electron microscopy is a very advance and powerful means of observing changes in a material post irradiation.

Main two types of electron microscopy are scanning electron microscopy (SEM) and transmission electron microscopy (TEM). As the name describes the basic microscope operation and design however there are multiple different detectors which can be fitted to both of these instruments which may enhance their characterization abilities.

To study a material's microstructure post radiation damage and even longer for general microstructural studies this instruments are of much needed tools. A TEM can give visual insight into the mechanisms of damage. It is a complimentary technique that like many other macroscopic techniques does have its limitations. Limitation are related to sample size and sample preparation, It is critical and will also influence what is seen in the instrument i.e small and thin samples are processed. This can leads to artefacts due to the size of the sample not being representative of the bulky material. The resolution of the TEM is very high, less than 1nm in an ideal sample although it does have its limits that may affect the ability to observe very small defect clusters. [13].

Use of TEM in studying radiation damage in material both pre and post irradiation in the literature is an extremely complex area. We will have some of the methods in this review.

The most commonly used methods in radiation damage studies is contrast mechanism arise from local changes in diffracting conditions caused by the effect on the defect strain field. This diffraction condition is can be divided into following two beam dynamical conditions, bright field kinematical, weak beam dark field and phase contrast [13] C.A. English [14] used a series of TEM contrast images to show the irradiation damage structure induced in pure metals and alloys by low-dose fission neutron irradiation at a range of temperatures. The use of this technique was able to show the different damage mechanisms and amount of

damage which occurs between the different crystal structures present in the pure metals and alloys.

Another key area of research where TEM may be utilized is the Characterization of dislocation loops and stacking fault tetrahedron (SFT) Defects in the 1 nm range are visible using the contrast mechanisms mentioned above, however to fully characterize these defects in the TEM we are limited by the resolution of the instrument as typically defects less than 5 nm are difficult. The full characterization can be divided in two areas. Distinguishing defect between dislocation loops, SFTs and small precipitates, then defining the nature of clusters as vacancy or interstitial.

Other area is loop Burgers vectors (direction) habit planes and shapes, measuring cluster sizes and distributions and the determination of number densities [13]. This was reported as being due to the lower loop mobility in Fe-Cr alloys as a result of pinning by Cr atoms. TEM is also used for characterization of voids and bubbles as a result of radiation damage. Voids in the range of 5nm or larger may be visible using the contrast mechanism discussed above. This can be achieved with manipulating with samples like sample tilts under focus, over focus condition which gives rise to white dots surrounded by black fringes. These images are analyzed. Calculations are applied to these images to measure the size of voids or cavities but because of very small size of less than 2nm accurate estimation is difficult [5]. Through focus technique to characterize two grades of martensitic commercial steels alongside an oxide dispersion strengthened ferrite alloy post irradiation.

Another instrument which is widely used in research is scanning electron microscope (SEM). This device famous for its capability to look at large samples without extensive need to prepare the samples unlike TEM. The SEM is considered a complimentary technique to TEM. We will study some examples on how this instrument is used. SEM has a lower resolution than the TEM although modern field emission gun based SEMs have come a long way to closing this gap in spatial resolution. Due to this resolution limit the SEM is more commonly used for bulk techniques rather than trying to image very small defects in the material. These bulk techniques include wavelength and energy dispersive spectroscopy (WDS, EDS) as a means of studying bulk chemical compositions and electron backscatter diffraction (EBSD) which is typically used to study any changes in a material's mechanical property at a microstructural level as a result of radiation damage.

Energy dispersive spectroscopy (EDS) technique has been used to study corrosion behaviour of Inconel alloy 800 under simulated supercritical water conditions. Results obtained from this study using EDS in the SEM were able to show how the elemental concentration distribution coupled with the morphology of the corrosion samples formed two distinct layers. An outer iron enriched layer and an inner layer enriched with chromium and nickel oxides. These layers

showed differing degrees of porosity for the different exposure temperatures that ultimately lead to a higher degree of porosity and therefore lower corrosion resistance for the samples exposed at higher temperatures.

As discussed earlier we can use various devices with this system so use of EBSD in an SEM as a means of studying flow localization in candidate structural materials for Gen-4 reactor systems. As stainless steel 316L is sensitive to irradiation damage in the temperature range of 150-400°C even at very low doses causing a severe loss of ductility. EBSD has been used to study the mechanism for which this loss of ductility occurs under these conditions.

Scanning transmission electron (STEM) detector fitted with a SEM may be used with good results for high-resolution imaging if the funds for dedicated TEM are not available. But samples preparation requirements for this are the same high standards as TEM

It should also be noted that Energy dispersive X-ray spectroscopy (EDS) is also an important analysis technique that may be utilized in the TEM. The main advantage to perform this type of analysis in the TEM is the very small probe size and x-ray interaction technique to show how radiation-induced grain boundary segregation differs in two grades of 304 stainless steel post neutron irradiation. It was shown that certain elements would segregate giving rise to higher inter granular stress corrosion cracking tolerance for one grade leading to a significant effect on the material's mechanical properties. Further work using x-ray analysis was performed to determine the origins of irradiation-assisted stress corrosion cracking in austenitic alloys in light water reactors. This work was performed using a proton beam instead of neutron, however, similar conclusions were made with respect to the segregation of certain elements at the grain boundaries.

#### 4. CONCLUSIONS

Radiation damage mechanisms are due to the impact of Fast neutron on metals, leads voids formations causes hardening. Also the helium production causes the embrittlement in metals. The purpose of this paper is to gain understanding of radiation damage mechanisms and their effect on system and its components in nuclear reactor.

Also the radiation characterization techniques Ion beam irradiation and Neutron irradiations along with TEM, SEM.

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