Synopsis
Recently, it has been found that CANDU garter spring made of Inconel® X-750 become embrittled after long time reactor exposure. To emulate the neutron irradiation induced phase and microstructural changes and understand the problems, in-situ heavy ions irradiations were performed. In this study, it is unveiled that irradiation induced phase change, lattice defects and cavities all contribute to spacer embrittlement. Dynamic formation of stacking fault tetrahedras (SFTs) by irradiation was first time ever observed. Through heavy ions irradiations combined with pre-implanted helium, we find that helium interstitials are essential for the cavities formation.

Introduction
Inconel® X-750 is a γ’ Ni3[Al, Ti] precipitation strengthened Ni based superalloy used for fastners and centering pins in the cores of pressurized water reactors and boiling water reactors, and in CANDU fuel channels as a spacer material. In-reactor neutron irradiation causes damage by cascades and reaction products resulting in formation of loops, cavities, and other defects, which will strongly affect the mechanical properties. There is concern in the industry that the springs may become embrittled after long reactor exposures and no longer function properly. To study the neutron irradiation damage, TEM in-situ heavy ions irradiation is performed. We benefit from in-situ heavy ion irradiation because: 1. no radioactive production, 2. more accurate parameters, 3. high dose rate, 4. dynamical observation, 5. cost effective.

Methods and Results
Samples were cut from a ready-for-service garter spring, ground and twin-jet electropolished for TEM observation. Samples were then irradiated by 1 MeV Kr++ ions at 60°C, 200°C, 300°C, 400°C, 500°C, and 600°C respectively up to 5.4 dpa in an intermediate voltage electron microscope (IVEM). Microstructures evolution and phases changes were observed at several dose steps. Post-irradiation EDX-STEM mapping was employed to analyze the phase change of γ’. Two beam bright field (TBBF) dynamical and kinematical conditions, weak beam dark field (WBDF) imaging and gb analyses were used to characterize defects. In order to emulate the in reactor neutron irradiation, which produces helium interstitials by a (n, α) transmutation reaction, in-situ heavy ion (Kr++) irradiations with pre-injected helium were performed under observation of IVEM.

Irradiation Induced Phase Change
While irradiated at 60°C~400°C, γ’ superlattice reflections disappeared at dose ~0.06 dpa, indicating the γ’ precipitates were totally disordered. Post irradiation EDX-STEM mapping indicates γ’ were not dissolved at this level. At higher dose 5.4 dpa, mapping shows a blurred edge of γ’ precipitates, indicating they are probably going to be dissolved at higher dose, in terms of competition between ballistic mixing and thermal ageing. γ’ precipitates were found stable at irradiation temperature >500°C up to 5.4 dpa. A critical temperature was found in between 400°C and 500°C, where irradiation effects and thermal ageing reaches the equilibrium point. Carbides were found stable at all temperature up to 5.4 dpa.

Irradiation Induced Lattice Defects
TBBF and WBDF observations were carried by using g=200 close to zone axis <110>. Defects consist mostly of SFTs, ½<110> perfect loops. ½<111>Frank loops formed only at temperature >500°C. Fractions of SFTs and loops are neither temperature nor dose dependent. There is no defects size change with dose, except evolution of Frank loops at elevated temperature. Defects sizes increase with temperature due to the mobility increase of interstitials and vacancies. SFTs formed directly from cascade and number density only depends on the ions flux. Measurement of defect density shows a great increase at low dose and saturation after 0.68 dpa due to damage overlapping.

Irradiation Induced Cavities
There are NO VOIDS found from sample irradiated by Kr++ even at high temperature 600°C, indicating the transmutation produced helium may be significant in swelling. As a result, the following experiments were performed. 200 appm He+ (50 keV) were pre-implanted within TEM, and then followed by 1 MeV Kr++ in-situ irradiation to 20 dpa. After helium pre-implantation to 200 appm at RT, there are no visible implantation induced defects and no helium bubbles. After Kr++ irradiation, however, cavities comparable to those in ex-service spacers, with sizes smaller than 5 nm, were observed by using TBBF kinematical condition.

Conclusions
The X-750 spacers lose strength and hardness due to disordering of the principal strengthening phase γ’. Disordering and dissolving of γ’ at reactor temperature will enhance swelling because fine γ’ precipitates are supposed to suppress cavities growth. Large amounts of SFTs and loops can harden the material at even low dose (0.7 dpa). Helium interstitials are essential for nucleation and stabilization of irradiation induced cavities. Those cavities play a vital role in GB embrittlement of the material. In this study, irradiation environment of CANDU reactor was successfully emulated by using heavy ion irradiation with pre-implanted helium. Future work needs to be done to optimize the material.