Re-examination of ion irradiation as a credible tool to simulate high energy neutron and proton-induced void swelling for accelerator-driven devices

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IWSMT-13 (2016) Chattanooga TN

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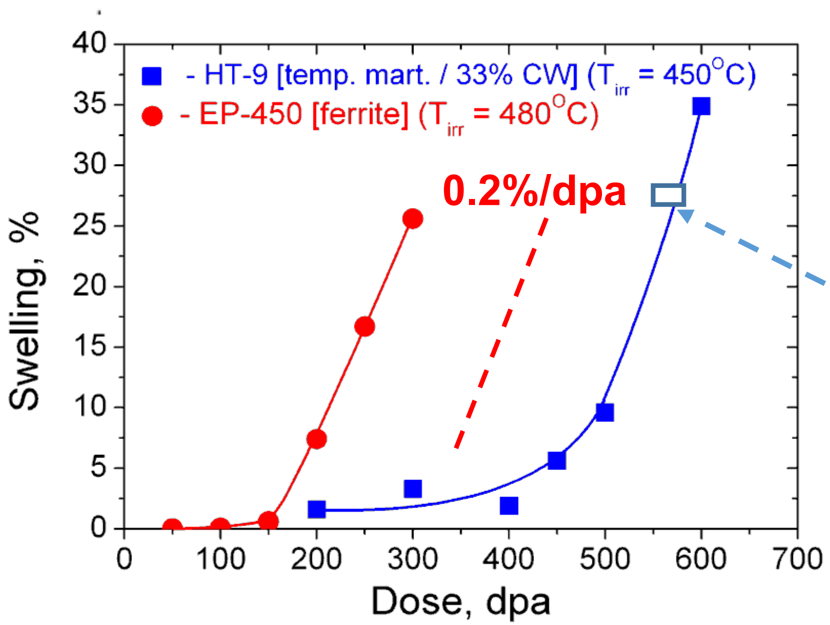
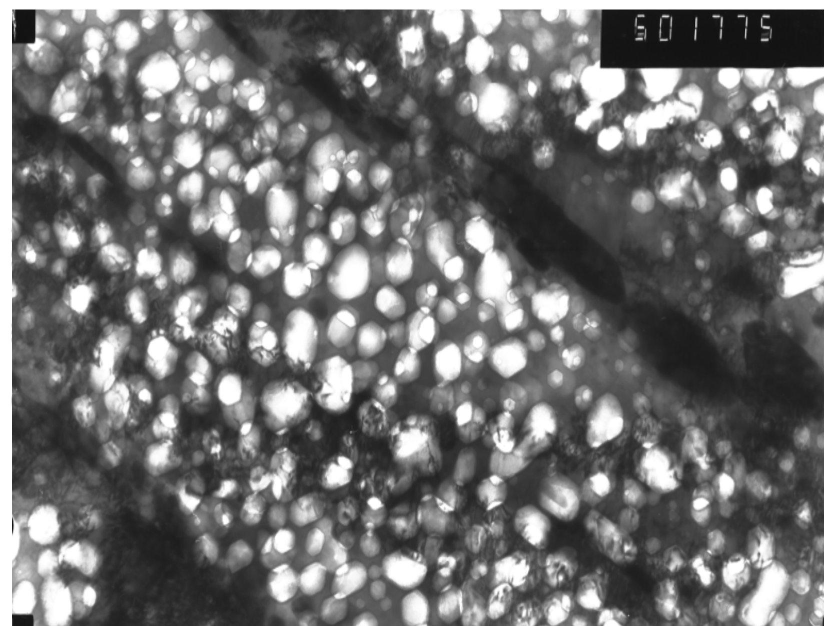
Credibility of ion simulation requires that ions reproduce major facets of neutron-induced behavior for both fcc and bcc iron-base alloys.

Recently conducted studies on bcc iron-base alloys appear to be successful in reproducing neutron-induced behavior

- **Linear after incubation swelling law**
- **Post-transient swelling rate of ~0.2%/dpa**
- **Increase in transient duration with increasing dpa rate**
- **Demonstrating strong influence of injected interstitial**



1.8 MeV Cr ion



28% swelling at 450°C and 550 dpa

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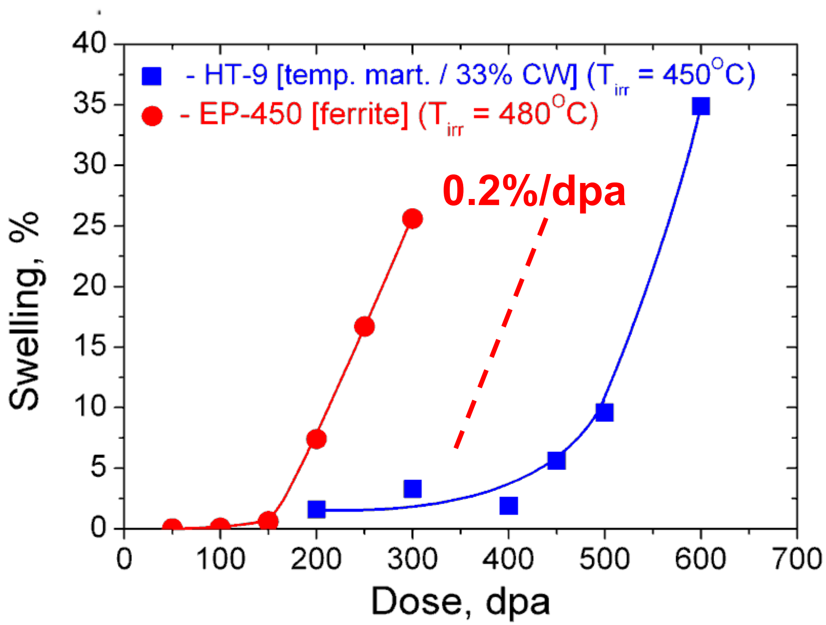

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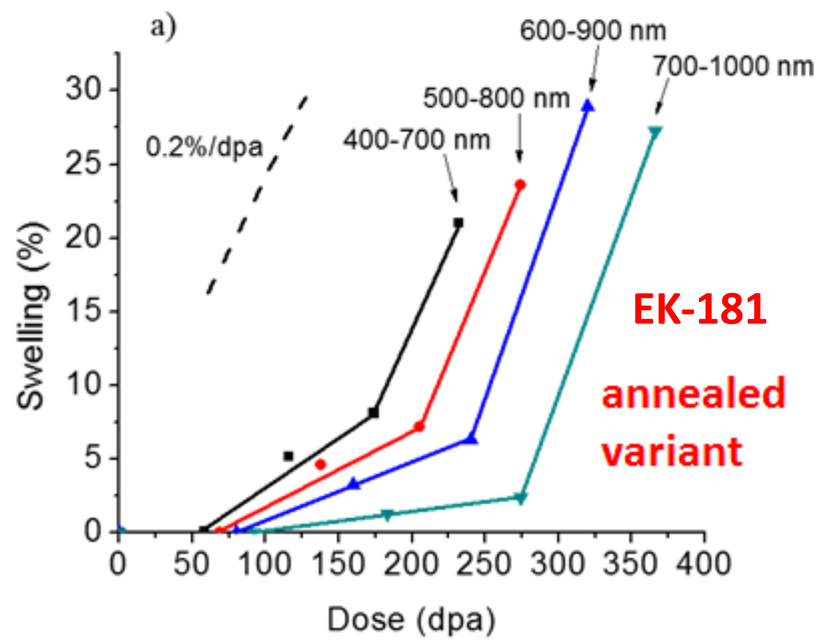

3.5 MeV Fe ion

Recently conducted studies on bcc iron-base alloys appear to be successful in reproducing neutron-induced behavior

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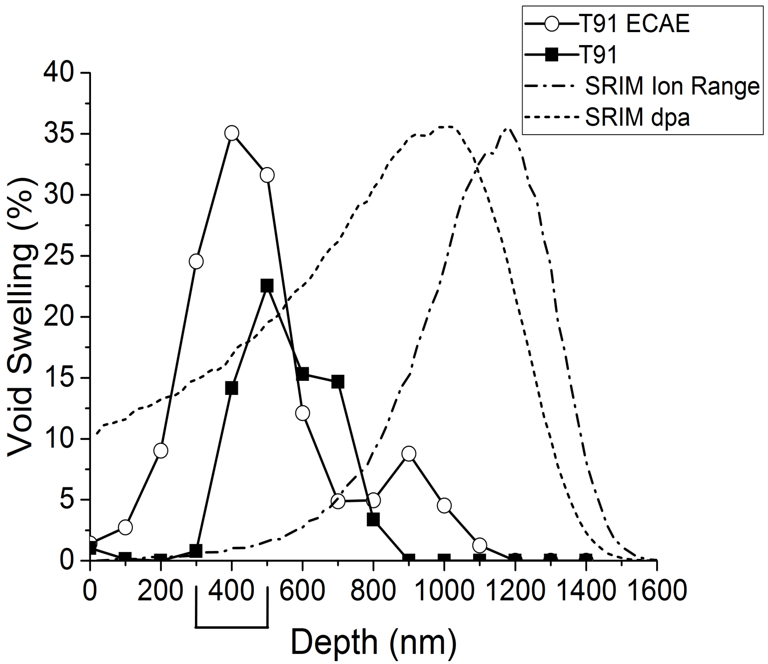

Dose profile and injected ion profile were calculated using SRIM computer code.

Injected ion
Injected interstitial

Injected interstitial effect leads to heavy suppression of void nucleation and measurable suppression of swelling rate if voids were produced before ion irradiation.

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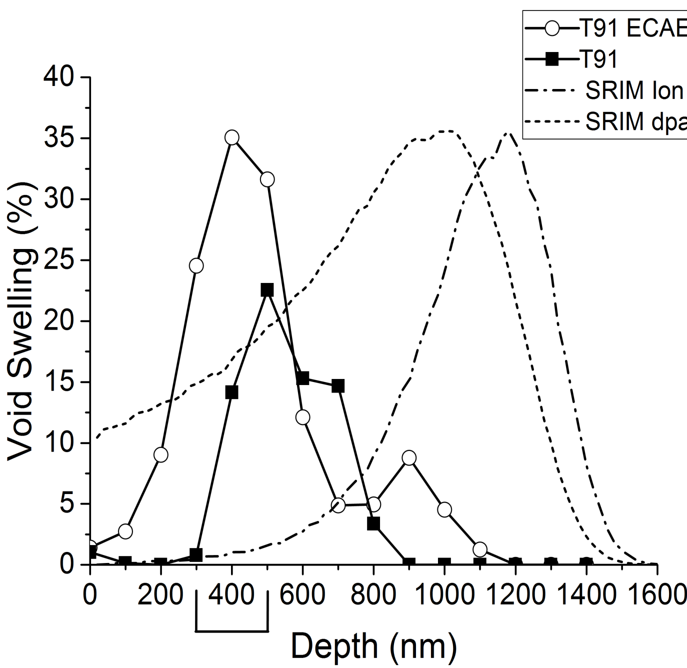

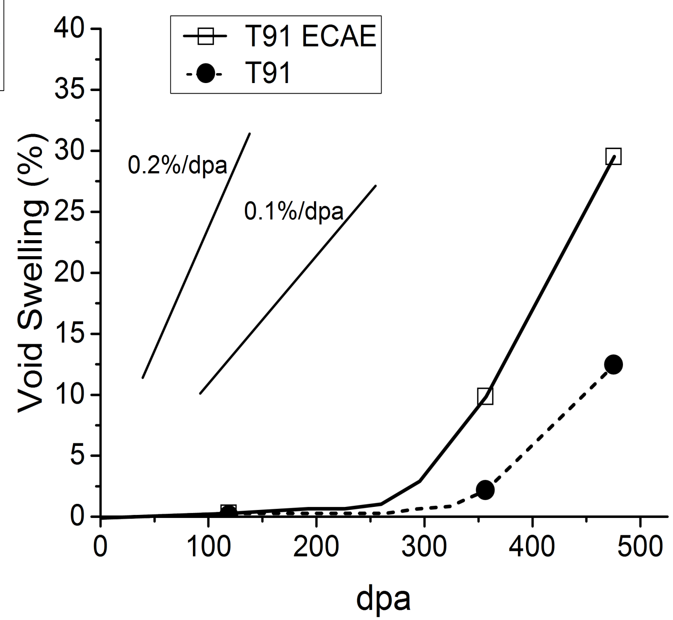

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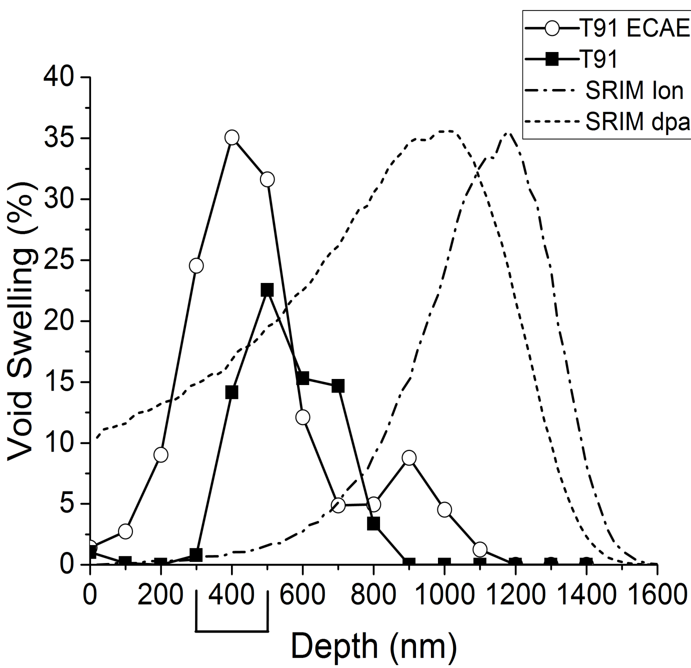

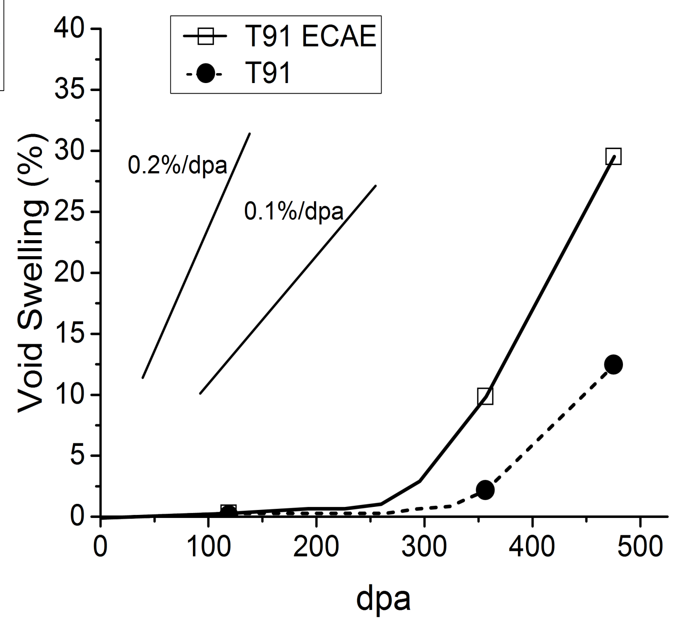

Was comparable success attained in earlier studies for fcc iron-base alloys?

- **This discussion is confined only to <u>self-ion</u> irradiation to simulate neutron-induced swelling.**

- **A tale of two generations of “ion bombardiers” and the <u>gulf</u> between them!**

- **My generation and …… the current generation**

- **Earlier groups led by Johnston, Kulcinski, Spitznagel, Bleiberg, Mansur, Packan-Farrell, Laidler ……….**

Some history concerning ion irradiation in USDOE National Laboratory programs

- **Void swelling at ~1% was discovered in U.K. in late 1960s.**
- **USA confirmed significant swelling (10-12%) in EBR-II.**
- **Large irradiation programs were initiated in EBR-II and FFTF.**
- **Neutron data was being accumulated in EBR-II at only 10-15 dpa every 18 months, mostly on fcc Fe-Cr-Ni alloys.**
- **Charged particle irradiations were employed to speed up data generation ……..1970s and 1980s, along with neutron-charged particle inter-correlation programs.**

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- **Charged particle irradiations were employed to speed up data generation ……..1970s and 1980s, along with neutron-charged particle inter-correlation programs.**
- **When neutron data became much more available, charged particle irradiation was deemphasized.**
- **During 1990s and early 2000s charged particle irradiation was mostly of university interest.**
- **In 2010s USDOE expressed new strong support of charged particle simulation, mostly on bcc Fe-Cr base alloys and their ODS variants.**

Something changed in the period when USDOE did not have a strong interest in charged particle simulation.

- **Inter-correlation programs had shown that self-ion irradiation produced the most reproducible results at high enough dpa levels to generate swelling.**
- **Calculation of dpa vs. depth profiles were provided in the early period (IONDOSE, BRICE, EDEP-1)**
- **In the later period dpa vs. depth curves were provided by TRIM and SRIM.**
- **All codes produce essentially identical results, but …**

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- **During the interim the input energy loss parameters were reevaluated, especially for mid-Z elements (Fe, Cr, Ni, Cu, V,……).**
- **The predicted energy deposition rates decreased by 22-33%.**
- **The ranges increased correspondingly and the dpa values mostly decreased.**

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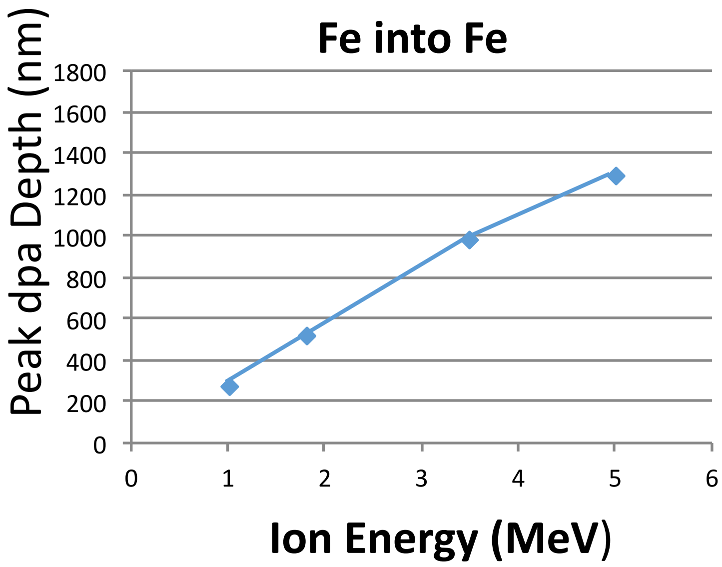
- **The predicted energy deposition rates decreased by 22-33%.**
- **The ranges increased correspondingly and the dpa values mostly decreased.**
- **The early experiments on fcc metals and fcc iron-base alloys …. and the perceptions that they produced were never reevaluated.**
- **As a consequence, the full impact of the injected interstitial in self-ion irradiation of fcc alloys was not fully appreciated.**

All of the experiments to be shown hereafter were performed before TRIM and SRIM were available.

There were a number of previous, independently produced codes (BRICE, EDEP, IONDOSE, etc.) and all required input parameters that had not yet been measured, but were calculated from the LSS model.

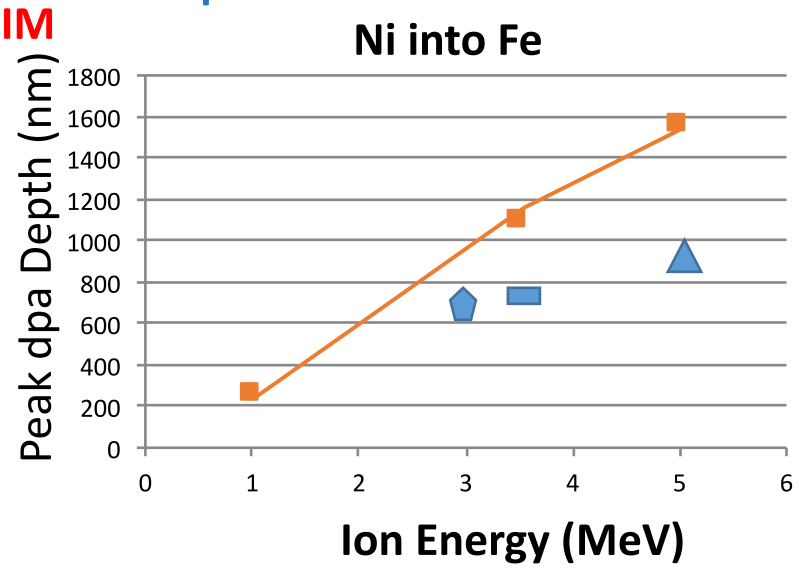
Note that all earlier cited studies used Ni ions, which travel farther than Fe ions at a given energy.

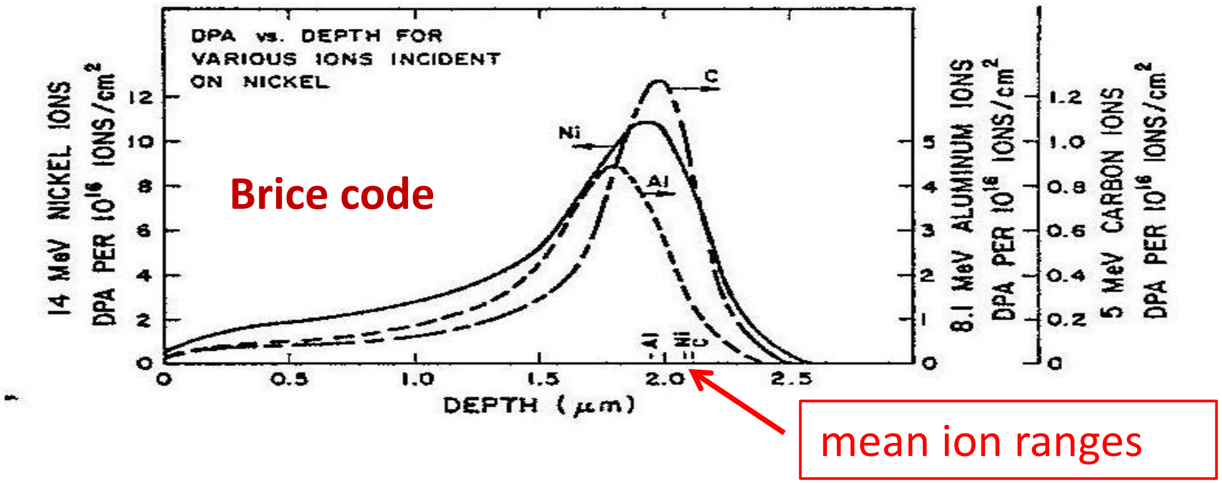
SRIM values of peak depth are much larger than EDEP values.



SRIM

Blue points are peak dpa positions used in earlier studies.

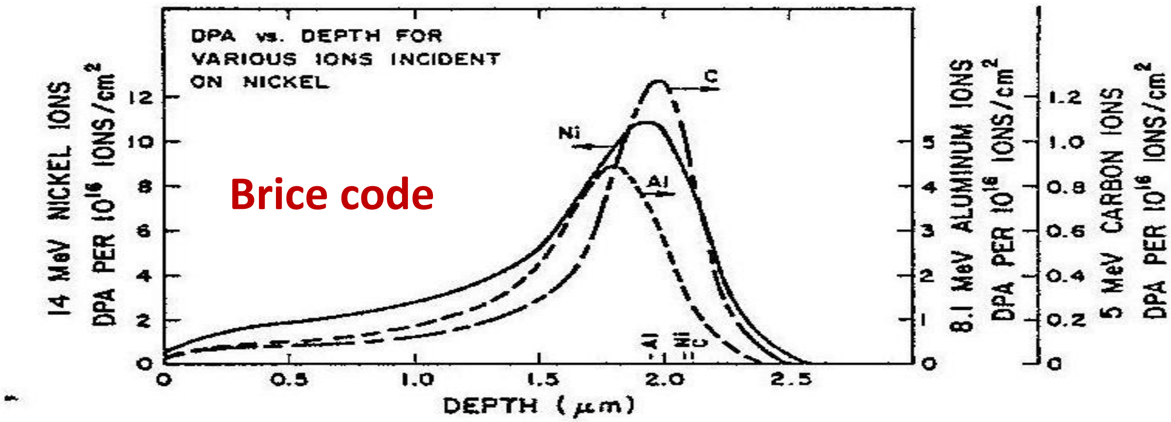




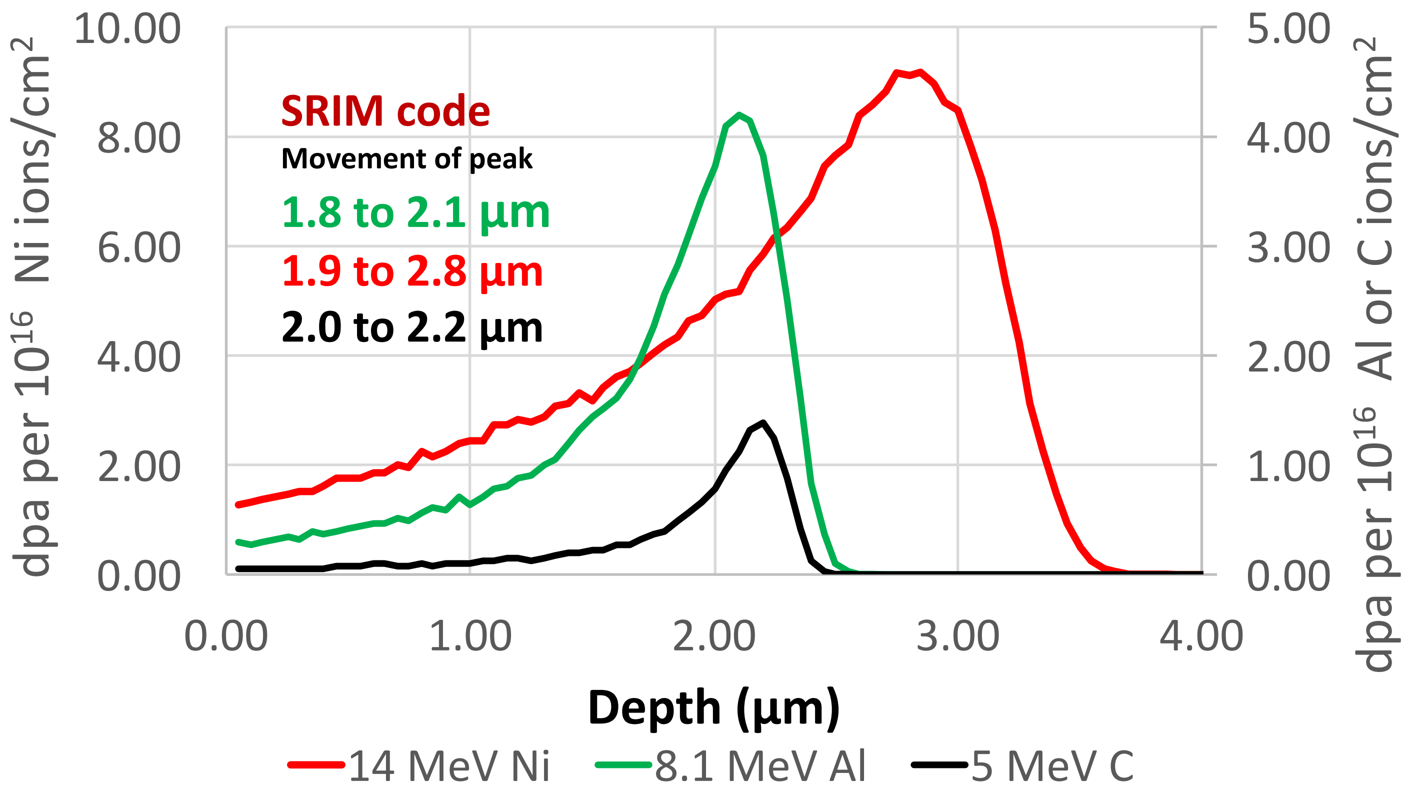
Dpa vs. depth curves for ion-irradiation of pure nickel

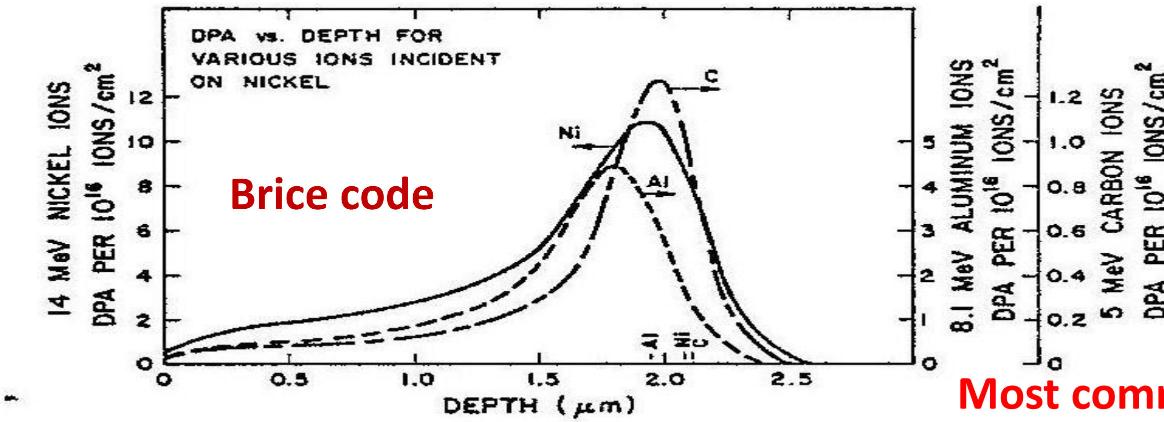
Calculated using the BRICE code for 3 different ions by J. B. Whitely of Kulcinski group at University of Wisconsin, 1979

14 MeV Ni, 8.1 MeV Al, 5 MeV C, all producing damage peaks at ~2 microns

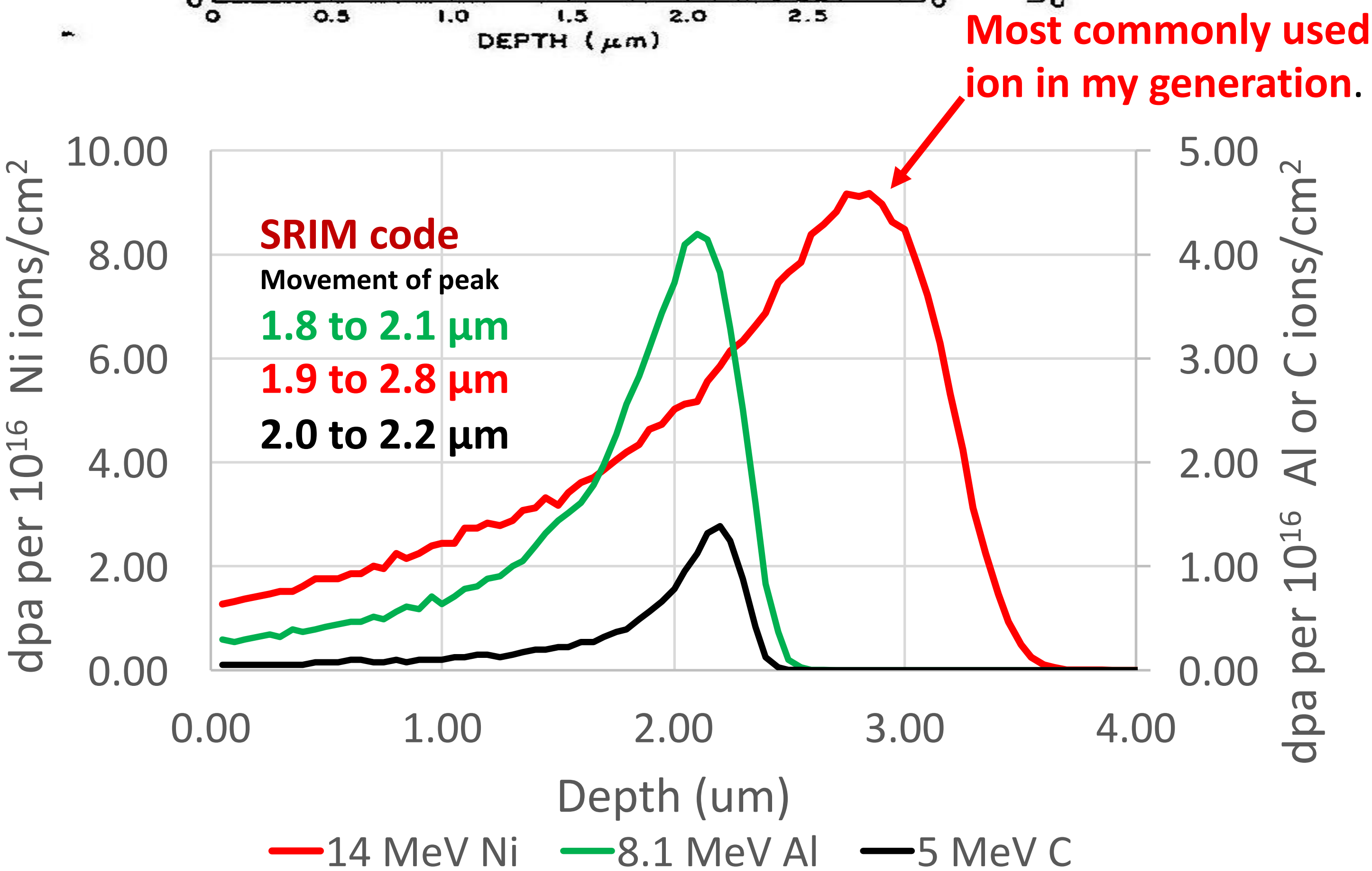


Note the relatively large change in the range of the nickel ion.





DPA vs. DEPTH FOR
VARIOUS IONS INCIDENT
ON NICKEL
Brice code
C
Ni
Al
14 MeV NICKEL IONS
DPA PER 10^16 IONS/cm^2
8.1 MeV ALUMINUM IONS
DPA PER 10^16 IONS/cm^2
5 MeV CARBON IONS
DPA PER 10^16 IONS/cm^2
DEPTH (μm)
0
0.5
1.0
1.5
2.0
2.5



Most commonly used
ion in my generation.
SRIM code
Movement of peak
1.8 to 2.1 μm
1.9 to 2.8 μm
2.0 to 2.2 μm
dpa per 10^16 Ni ions/cm^2
dpa per 10^16 Al or C ions/cm^2
Depth (um)
0.00
1.00
2.00
3.00
4.00
14 MeV Ni
8.1 MeV Al
5 MeV C

What criteria can we use to establish a credible relationship of ion simulation to neutron experience?

- **Does self-ion simulation reproduce the basic swelling law observed during neutron irradiation of iron-base alloys …. …… linear after incubation ("bilinear")**
- **Can ion simulation reproduce the post-transient steady-state swelling rates of**

  ~1%/dpa for fcc iron-base alloys

  ~0.2%/dpa for bcc iron-base alloys
- **Does self-ion irradiation reproduce the major trends of swelling with respect to major compositional, fabricational and environmental variables?**

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Let's look at the most famous example on the compositional dependence of Fe-Cr-Ni ternary alloys.

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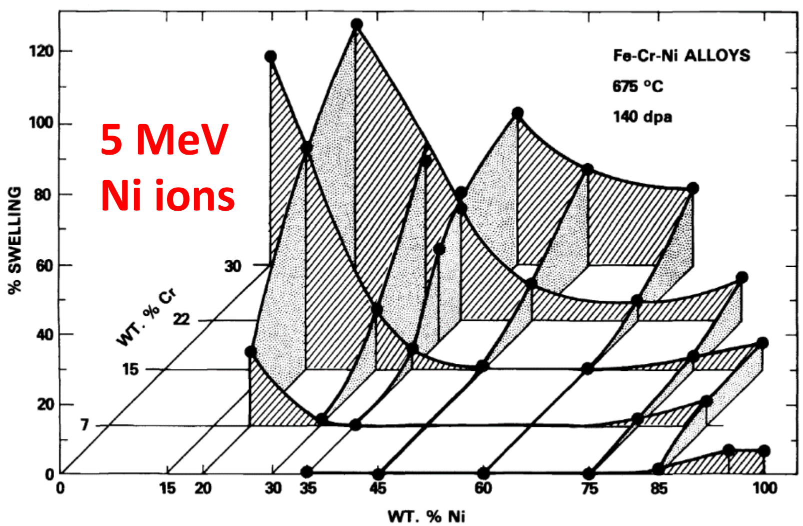
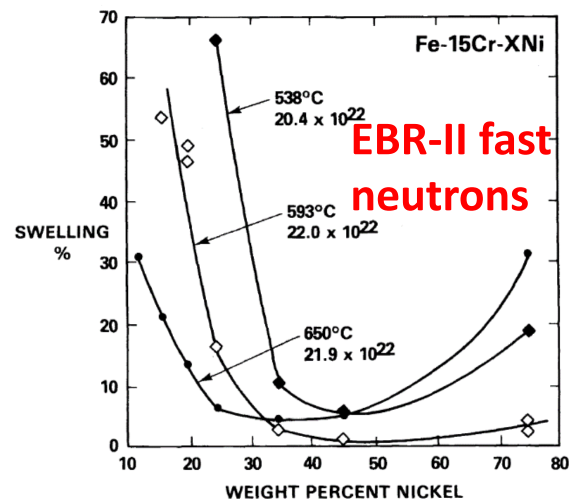


Johnston et al., 1983



Garner and Brager, 1988

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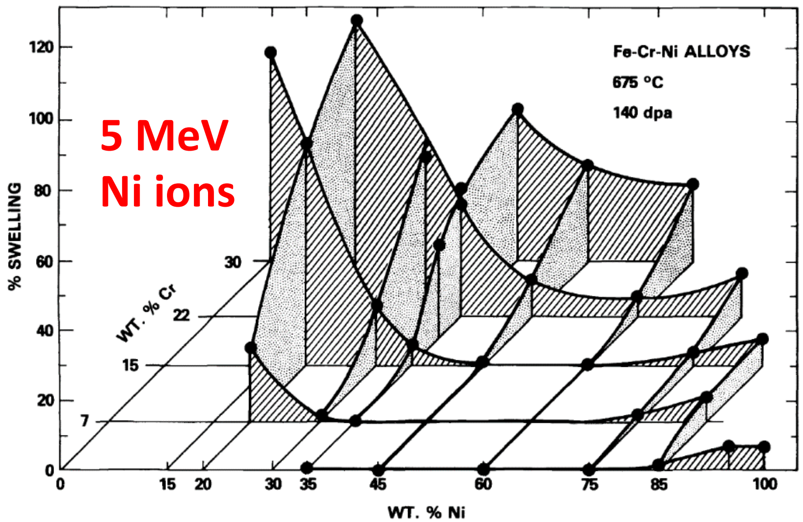


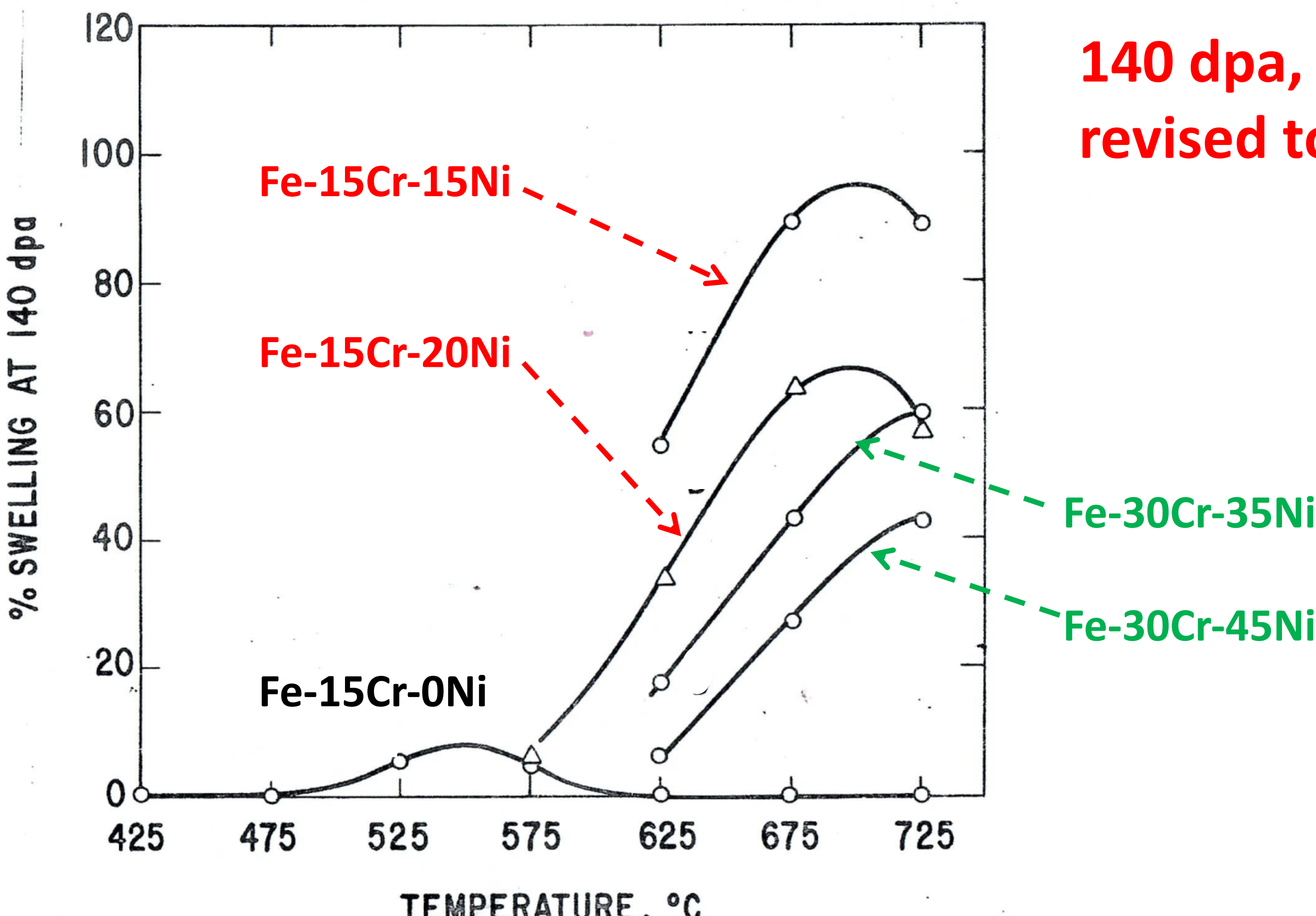
Johnston et al., 1983

Similar success for Cr, Si, P, cold-work, thermal treatment, etc. …… but my generation never clearly saw the 1%/dpa (fcc) or the 0.2%/dpa (bcc) in ion studies.

We also <u>never</u> saw a <u>convincingly clear</u> example of injected interstitial suppression.

Ferritic binary alloy swells during 5 MeV Ni^{+} irradiation at lower temperatures and at much lower rates than do ternary austenitic alloys

W.G. Johnston et al. , 1983

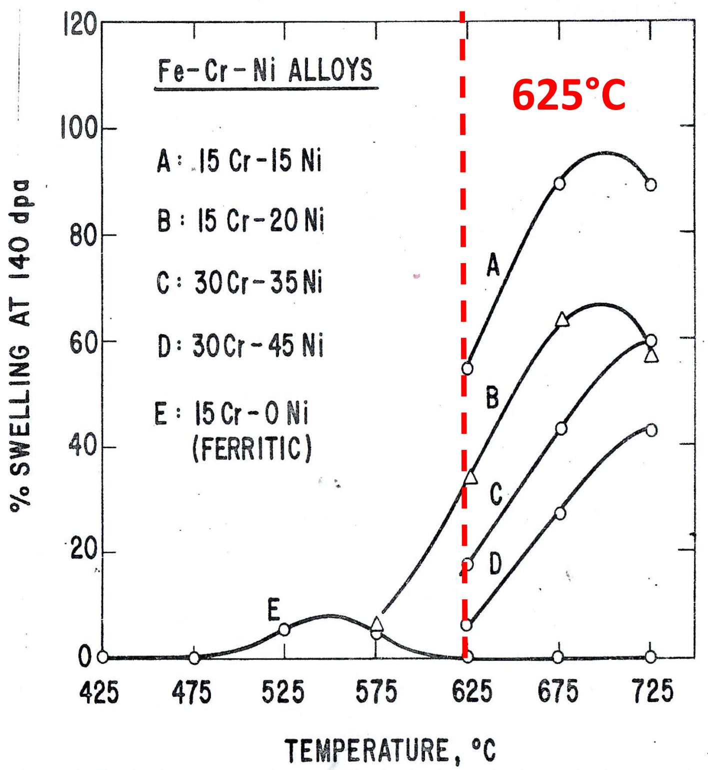


140 dpa, later revised to 116 dpa

Ion irradiation forecast the different swelling of bcc Fe-Cr and fcc Fe-Ni-Cr alloys

Johnston et al., 1983

140 dpa 5 MeV Ni^+ ions

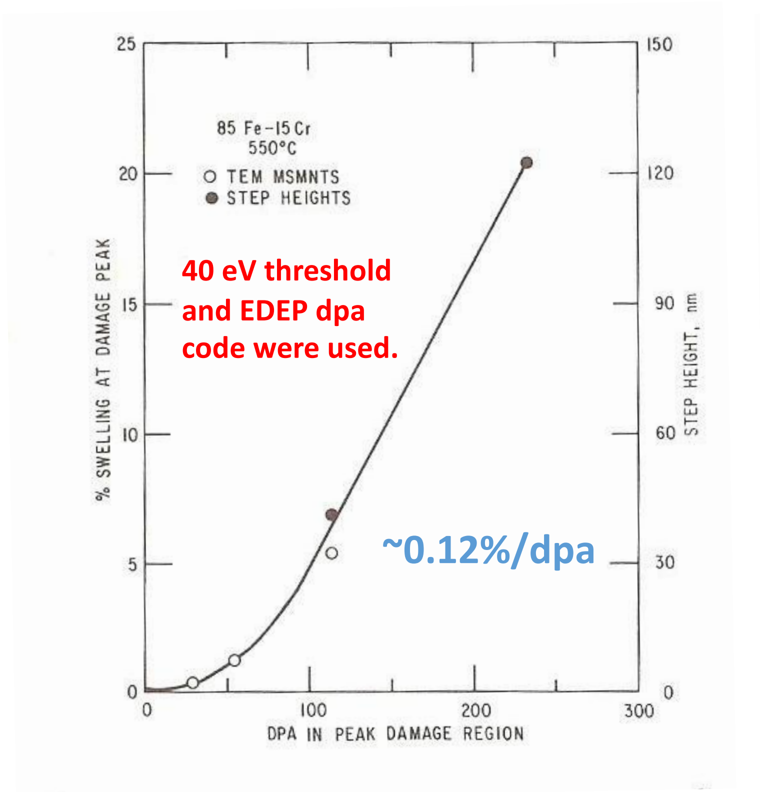


Surface of Uranus 50 duplex alloy irradiated at 625°C to 140 dpa

Ferrite grains swell less than austenite grains due to different swelling rate and different temperature regime of swelling

Swelling of Fe-15Cr model ferritic alloy at 550°C using 5 MeV Ni$^+$ ions

W. G. Johnston and coworkers, 1979



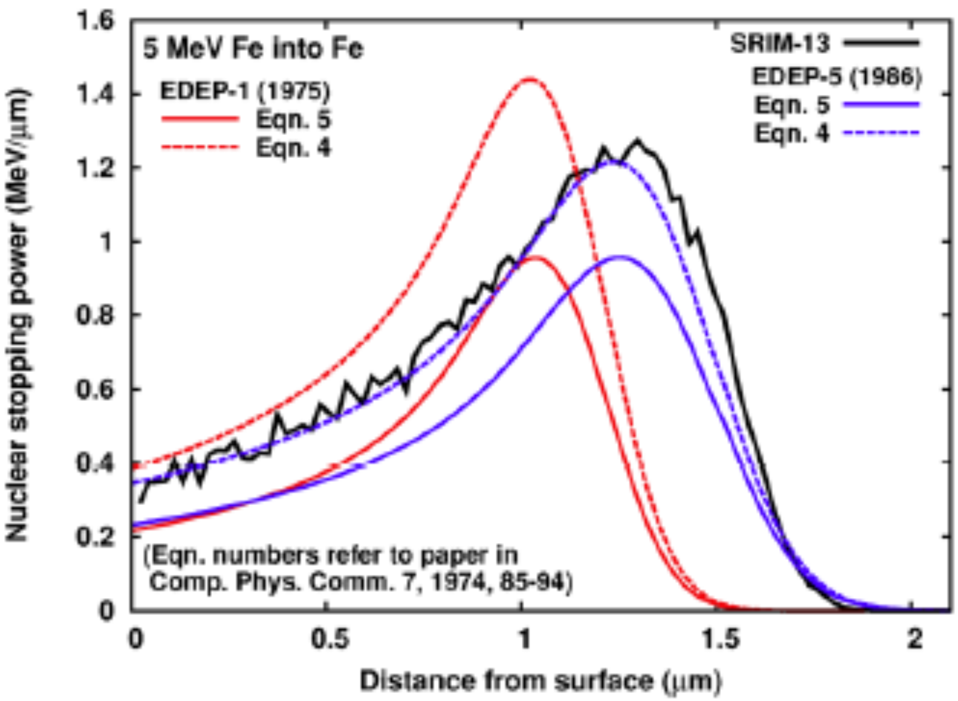
Johnston used both microscopy and step-height measurements.

Empirical value of ~60Å of height for each 1% swelling at the peak swelling position.

Swelling rate appears to be less than the expected value of ~0.2%/dpa.

SRIM-calculated doses would be much smaller and the swelling rate would be higher.

Comparison of ion-induced dpa predictions of EDEP and SRIM codes, courtesy of Roger Stoller, ORNL



Between 1975 and 1986 the EDEP predictions changed significantly.

1986 EDEP prediction of Eqn. 4 most closely matches SRIM-13 prediction.

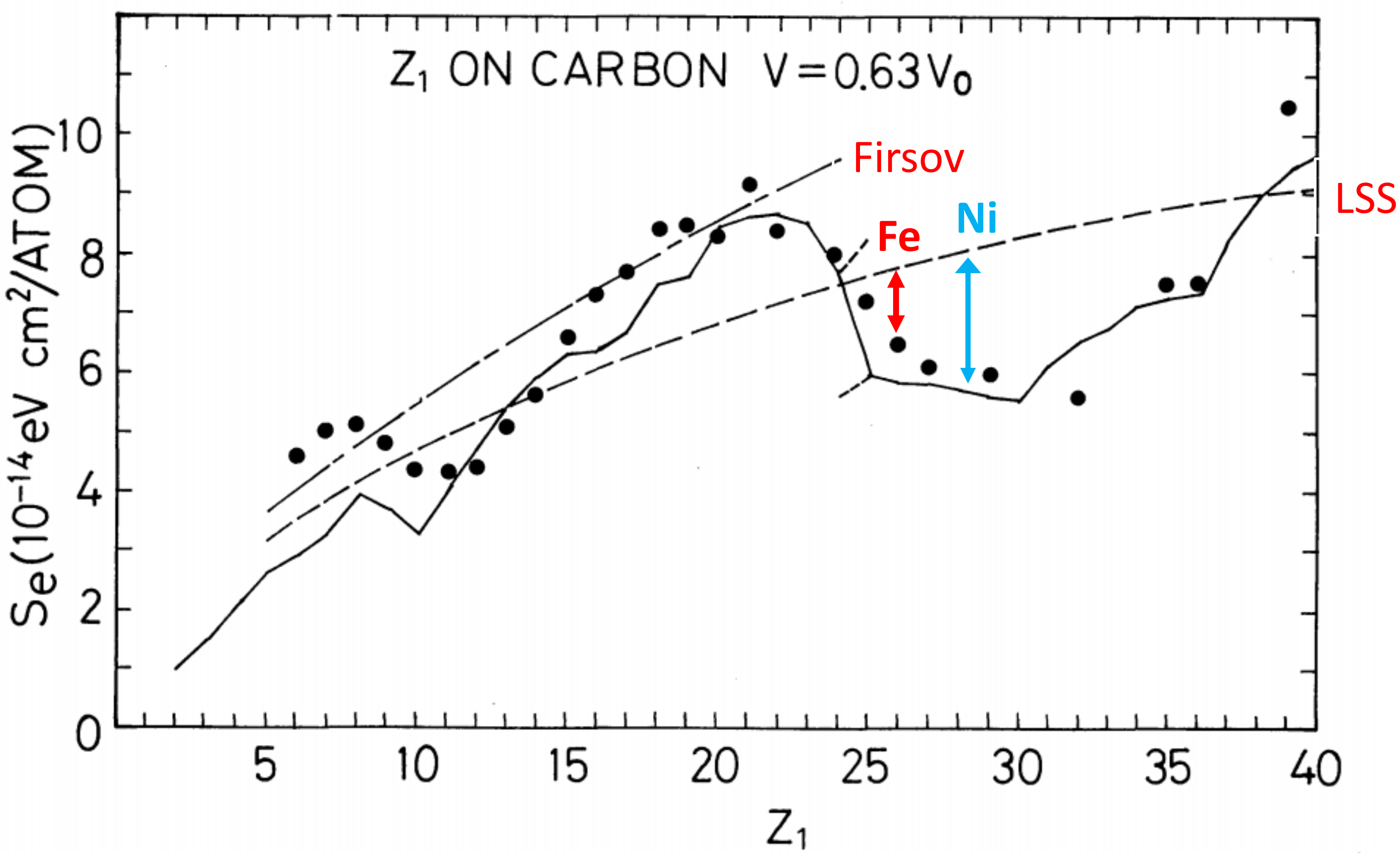
The difference arises not in the mechanics of each code, but in the energy loss equations that are input to the code.

SRIM uses Monte Carlo and takes a lot of time, but previous codes were much quicker and used analytical expressions, producing smooth curves.

What has changed in the energy loss descriptions over this time period?

Z_1 oscillation in electronic stopping power

Sugiyama, J. Phys. Soc. Japan, 50, 929 (1981)



Experimental data from Hvelplund and Fastrup, Phys. Rev. 165, 408 (1968)
LSS formula from Lindhard, Scharff and Schiott, K. Dan. Vidensk. Selsk. Mat. Fys. Medd, 33, 14 (1963)
Firsov formula from Firsov, Soviet Phys. JETP 9, 1076 (1959).

Stopping power oscillation

- Oscillation exists for both projectile (Z_1) and target (Z_2)
- Oscillation exists for both nuclear and electronic stopping powers
- Oscillation amplitude increases with increasing projectile energy
- Caused by quantum mechanics of the shell structure

- Atomic radius is oscillating with increasing Z
- Higher Z may have "smaller" atomic radius
- Smaller radius leads to higher electron screening effect
- Higher screening effect means less effective nuclear charge in collision

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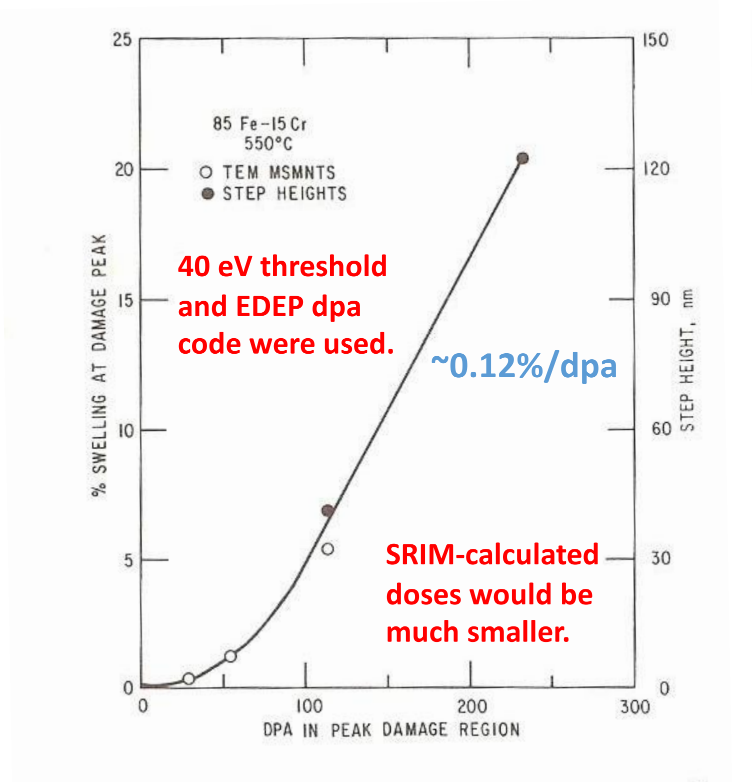
Swelling of Fe-15Cr model ferritic alloy at 550°C using 5 MeV Ni^+ ions

W. G. Johnston and coworkers, 1979



Johnston used both microscopy and step-height measurements.

Empirical value of ~60Å of height for each 1% swelling at the <u>peak swelling</u> position.

Swelling rate appears to be less than the expected value of ~0.2%/dpa.

Doses were recently reevaluated by Garner and Wang using SRIM instead of EDEP.

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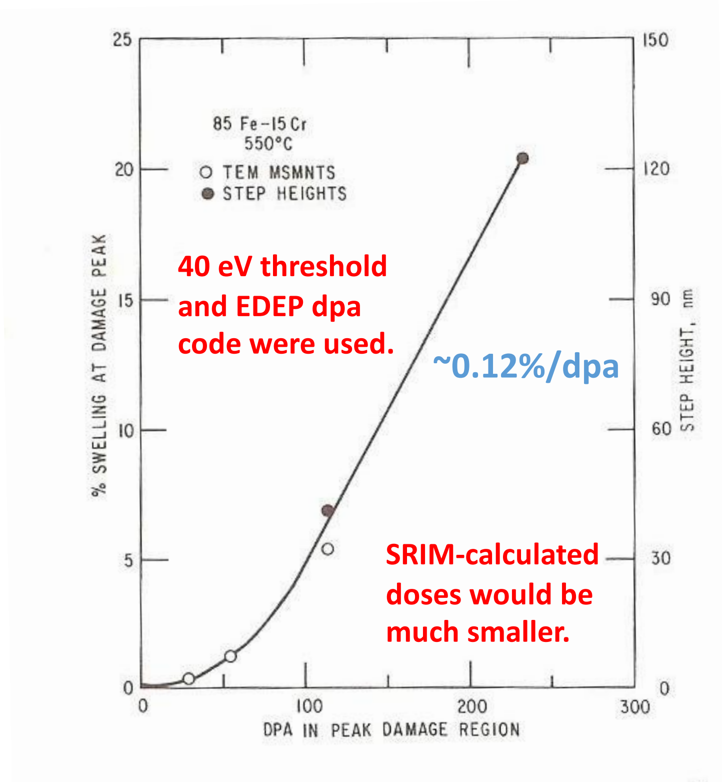
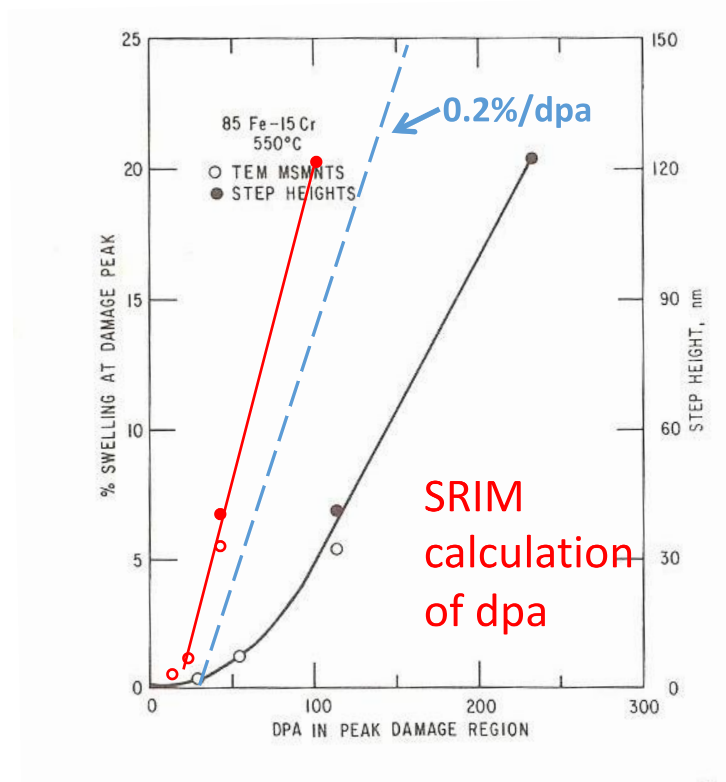
W. G. Johnston and coworkers, 1979





Use of SRIM-calculated doses shortens the transient regime of swelling and reproduces the ~0.2%/dpa swelling rate observed in neutron irradiations.

Let's re-examine four old data sets on depth-dependent swelling of fcc iron-base alloys in light of the new perception of dpa damage deposition curves.

All experiments used Ni ions

- **Annealed 321 SS at 5 MeV (GE)**

  Multiple thinning to reach different depths

- **Annealed Fe-15Cr-25Ni at 3.5 MeV (WARD)**

  HVEM and stereomicroscopy

- **20% cold-worked 316 at 5 MeV (WARD)**

  HVEM and stereomicroscopy

- **20% cold-worked 316 SS irradiated first in EBR-II (GE, ORNL)**

  and then further irradiated with Ni ions to higher dose at four higher temperatures.

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There are many more examples, all leading to the same conclusions concerning the increased credibility of older ion irradiation experiments.

Journal of Nuclear Materials 62 (1976) 167–180

Data were attained using a **multiple depth polishing technique** with some uncertainty on depths of observation.

THE DEPTH DISTRIBUTION OF VOID SWELLING PRODUCED BY 5 MeV Ni IONS

W.G. JOHNSTON, J.H. ROSOLOWSKI and A.M. TURKALO
General Electric Company, Corporate Research and Development, Schenectady, NY, USA

and

T. LAURITZEN
General Electric Company, Fast Breeder Reactor Department, Sunnyvale, CA, USA

Received 15 March 1976

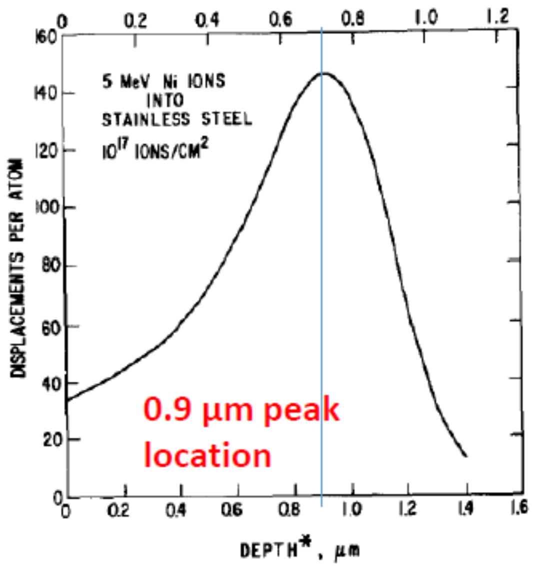


Fig. 1. Displacement profile calculated for 5 MeV Ni ions into stainless steel.

321 SS

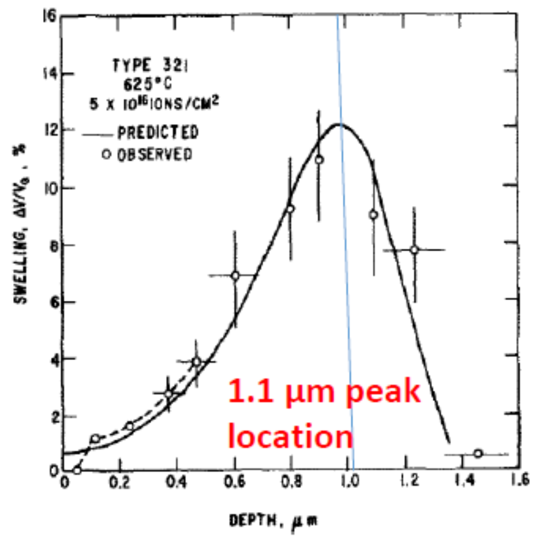


Fig. 6. Data points indicate the observed distribution of swelling in type 321 at 625°C, and the solid curve is the predicted swelling profile. Representative error bars are shown.

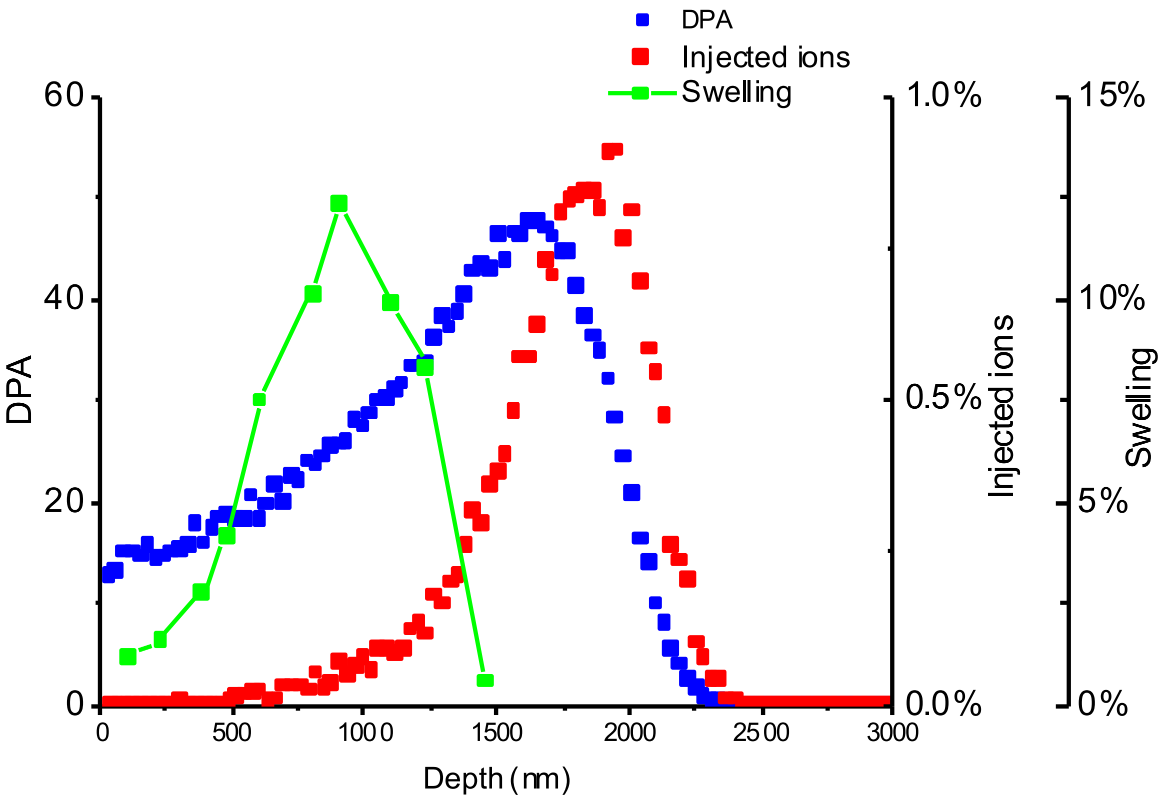
Dose vs. depth was calculated using EDEP-1 code in 1975.

Swelling peak is a little deeper than the dose peak.

There is no indication of the suppression of void nucleation toward the back of the range that is always observed in ferritic alloys.

5 MeV Nickel ions into 321 stainless steel

SRIM, KP option, 5x 10^{16} ions/cm^2 → new interpretation

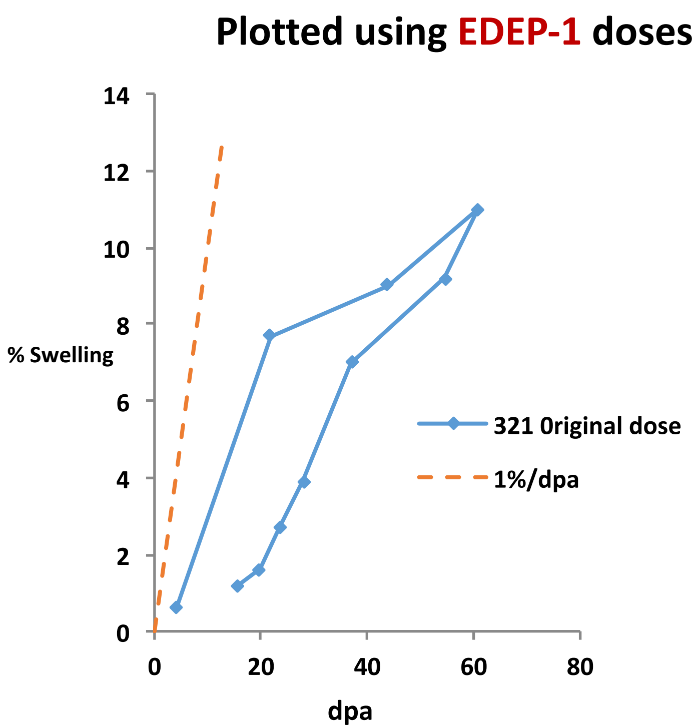


Injected interstitial suppression is very large, similar to behavior observed in ferritic alloys.

Swelling peak observed is only the "remnant peak " after severe suppression of swelling beyond ~1000 µm.

Note that the swelling peak does not coincide with the dpa peak as it did in the original interpretation!

If swelling is plotted vs. local dpa (EDEP-1), can we see the ~1%/dpa swelling rate that is characteristic of austenitic alloys?

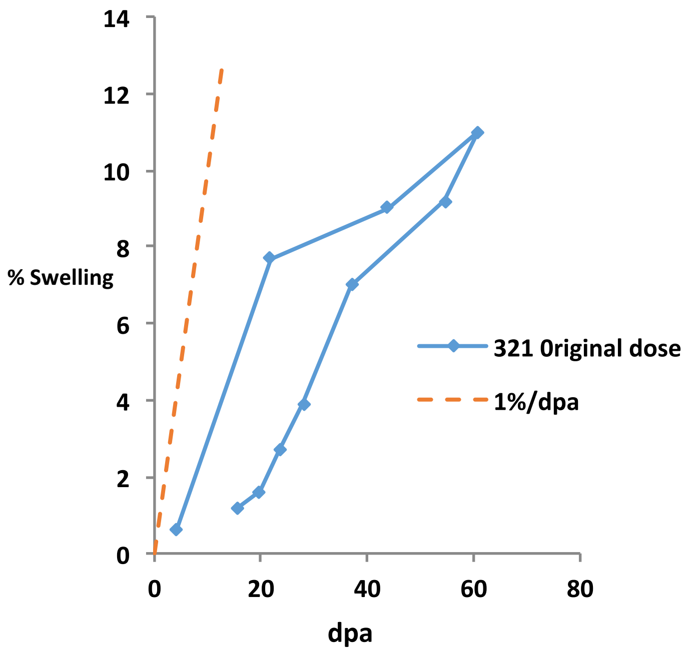


The swelling "loop" is an artifact of an incorrect dpa vs depth curve

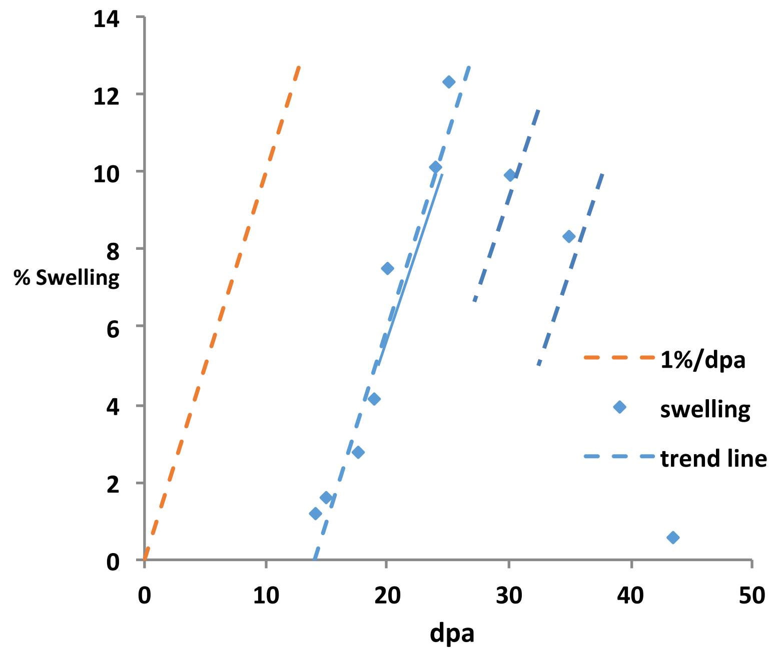
Swelling rate of ~1%/dpa is not observed on any portion of the loop.

If swelling is plotted vs. local dpa (EDEP-1), can we see the ~1%/dpa swelling rate that is characteristic of austenitic alloys?

Plotted using EDEP-1 doses



Plotted using SRIM doses



The characteristic swelling rate of ~1%/dpa is seen in the first seven measurements.

The three back-side measurements reflect the growing influence of injected interstitial and increasing dpa rate with increasing depth.

.

REFERENCE: Rowcliffe, A. F., Diamond, S., Bleiberg, M. L., Sptiznagel, J., and Choyke, J., "Swelling and Irradiation Induced Microstructural Changes in Nickel-Based Alloys," *Properties of Reactor Structural Alloys After Neutron or Particle Irradiation*, ASTM STP 570, American Society for Testing and Materials, 1975, pp. 565–583.

Swelling and dpa curve have peaks in close proximity.

No indication of injected interstitial suppression zone.

5 MeV Ni ion into 20%CW 316 SS

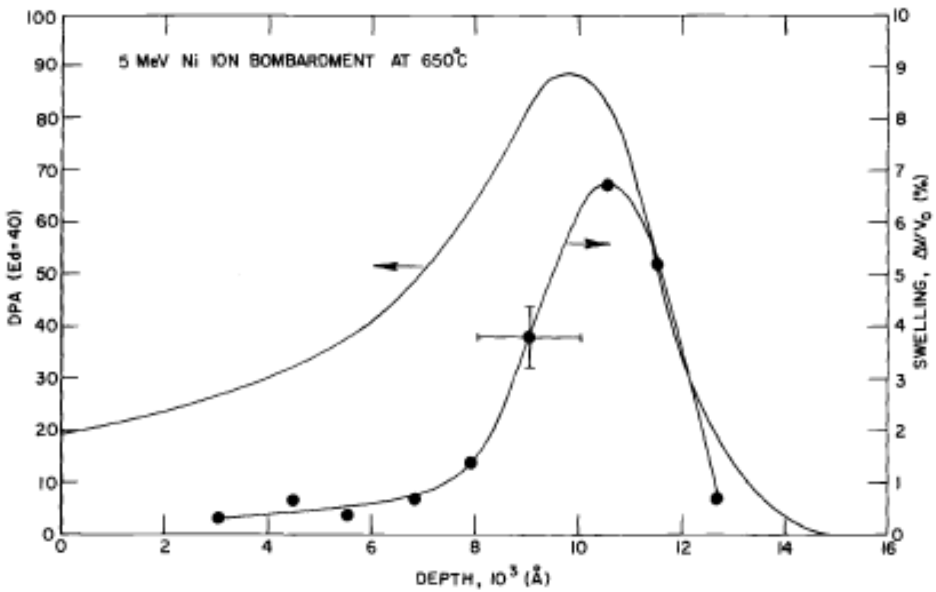


FIG. 7—Variation in displacement damage and swelling with depth below the bombardment surface for 20 percent cold-worked Type 316.

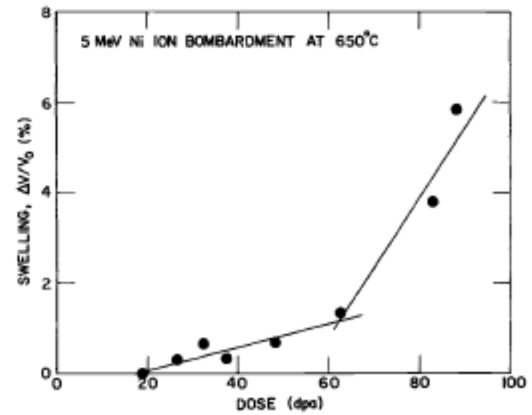


FIG. 8—Variation in swelling with dose for 20 percent cold-worked Type 316.

Extraction of swelling data used HVEM over entire ion range, reducing scatter and depth uncertainties.

REFERENCE: Rowcliffe, A. F., Diamond, S., Bleiberg, M. L., Sptiznagel, J., and Choyke, J., "**Swelling and Irradiation Induced Microstructural Changes in Nickel-Based Alloys,**" *Properties of Reactor Structural Alloys After Neutron or Particle Irradiation*, *ASTM STP 570*, American Society for Testing and Materials, 1975, pp. 565–583.

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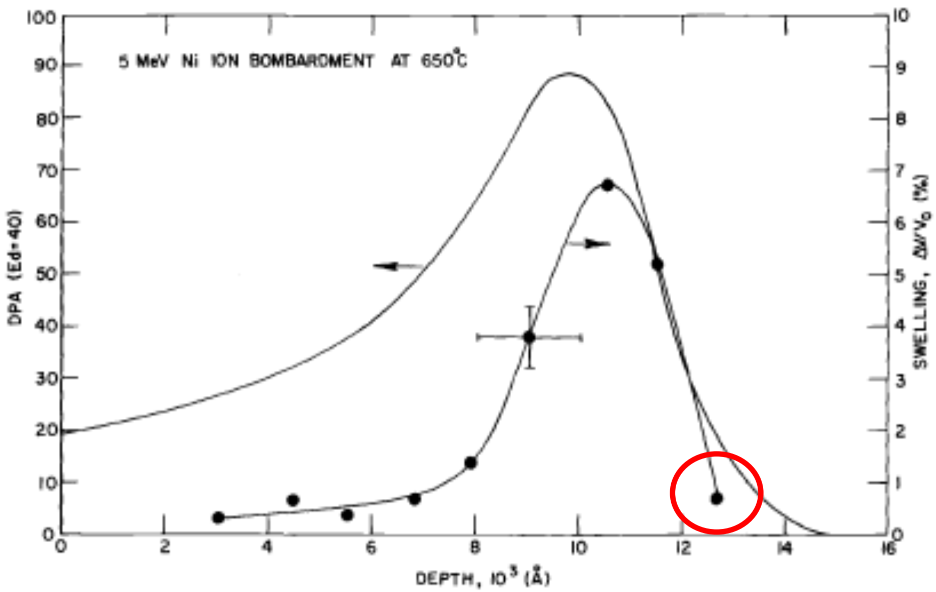


FIG. 7—*Variation in displacement damage and swelling with depth below the bombardment surface for 20 percent cold-worked Type 316.*

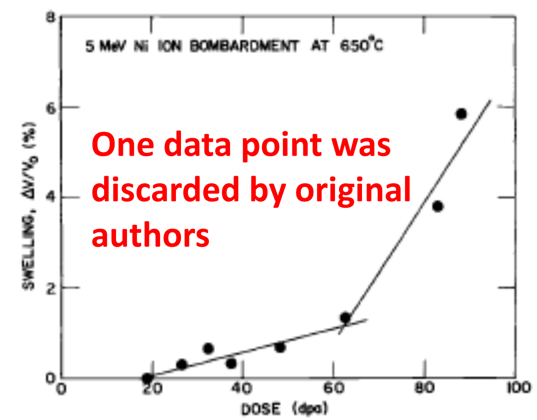
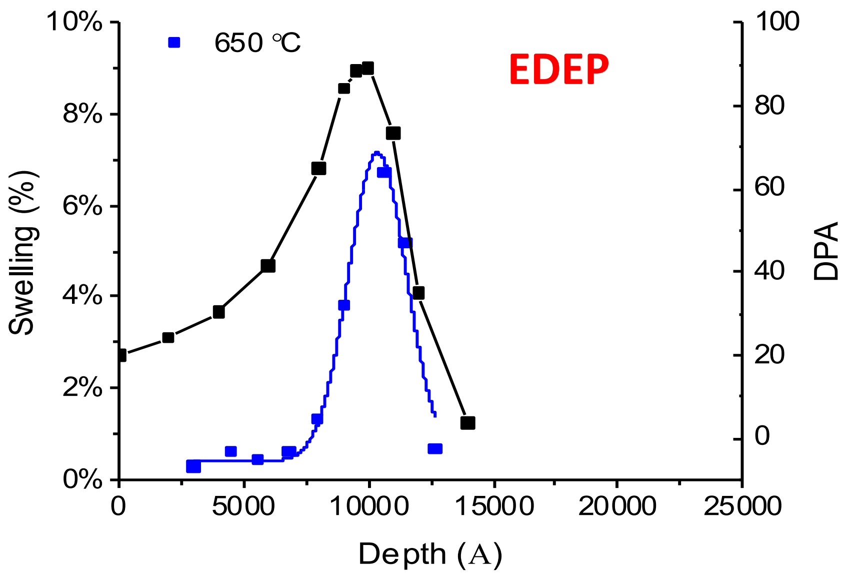


FIG. 8—*Variation in swelling with dose for 20 percent cold-worked Type 316.*

Extraction of swelling data used HVEM over entire ion range, reducing scatter and depth uncertainties.

Comparison: two interpretations of 5 MeV ion irradiation data on CW 316 stainless

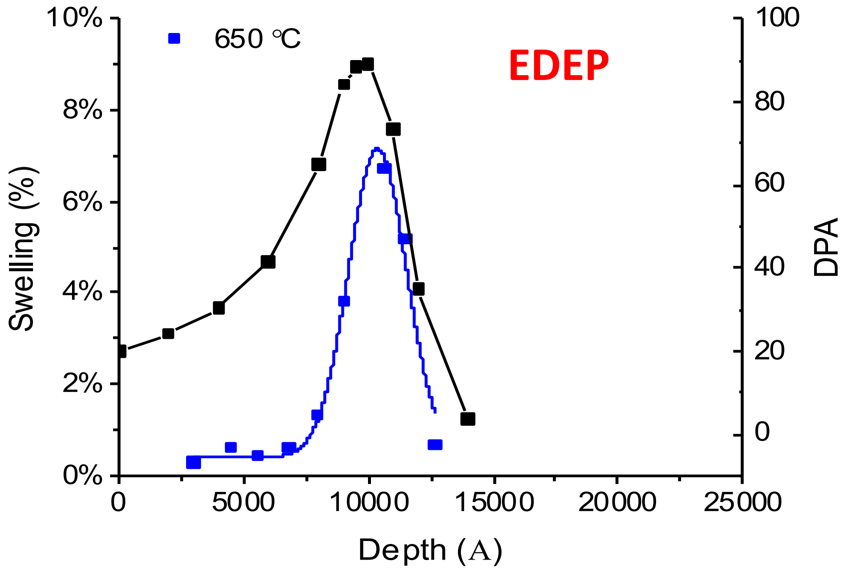


Peak damage dose of 90 dpa was calculated with EDEP to be ~1000 nm.

Swelling peaks at peak dose position.

Injected interstitial influence was either absent or was quickly overcome.

Comparison: two interpretations of 5 MeV ion irradiation data on CW 316 stainless



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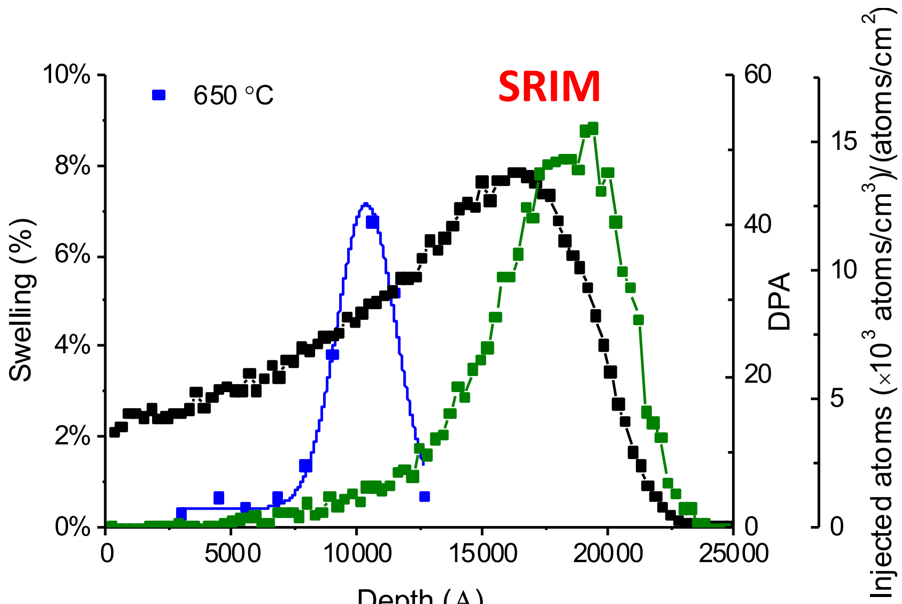
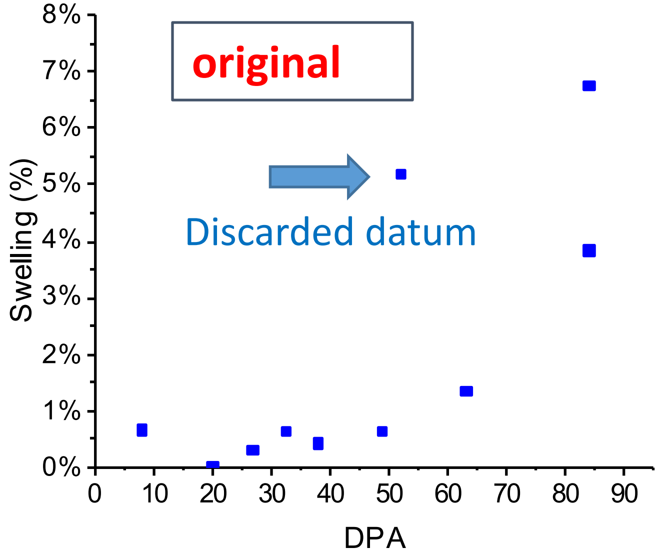


Peak damage dose was calculated with SRIM to be 47 dpa at ~1700 nm.

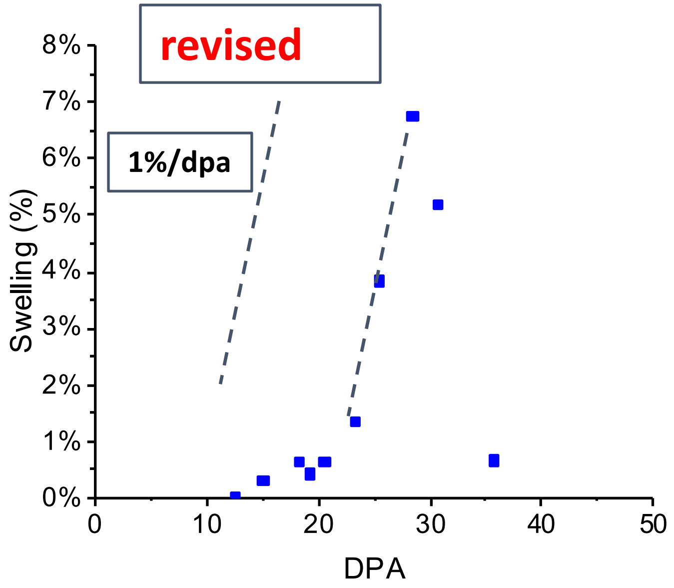
Injected interstitial effect is very strong to suppress void nucleation.

Assigned dpa levels drop strongly and post-transient swelling rates increase in region far from injected interstitial.

Comparison: two interpretations of 5 MeV ion irradiation data on CW 316 stainless

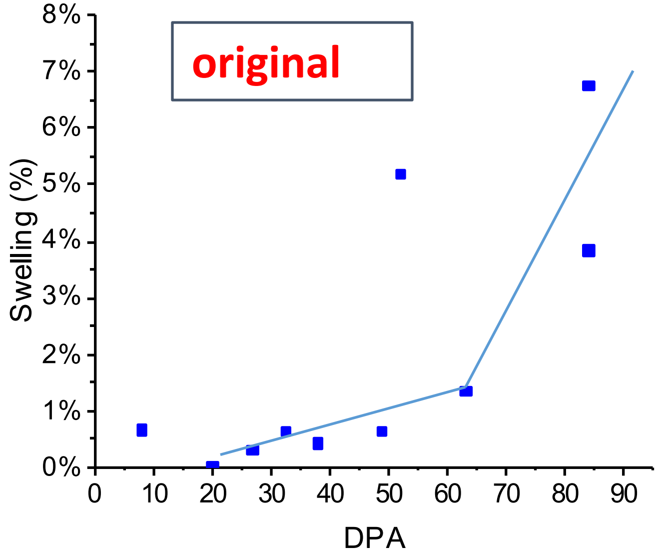


It is difficult to extract any estimate of local swelling rate from this plot.



Data taken far from injected interstitial implies a post-transient swelling rate of ~1%/dpa, similar to that observed in neutron irradiation.

Comparison: two interpretations of 5 MeV ion irradiation data on CW 316 stainless



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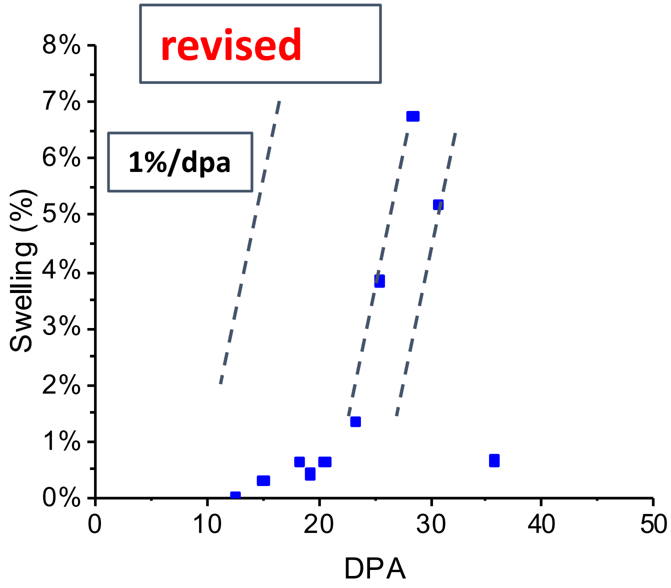

Data taken far from injected interstitial implies a post-transient swelling rate of ~1%/dpa, similar to that observed in neutron irradiation.

Beyond peak swelling shows increasing influence of injected interstitial and higher dpa rate.

Journal of Nuclear Materials 117 (1983) 177–197
North-Holland Publishing Company

IMPACT OF THE INJECTED INTERSTITIAL ON THE CORRELATION OF CHARGED PARTICLE AND NEUTRON-INDUCED RADIATION DAMAGE

F.A. GARNER

Westinghouse Hanford Co., P.O. Box 1970, Richland, WA 99352, USA

Swelling and dpa curve have peaks in close proximity.

No indication of injected suppression zone.

3.5 MeV Ni ions

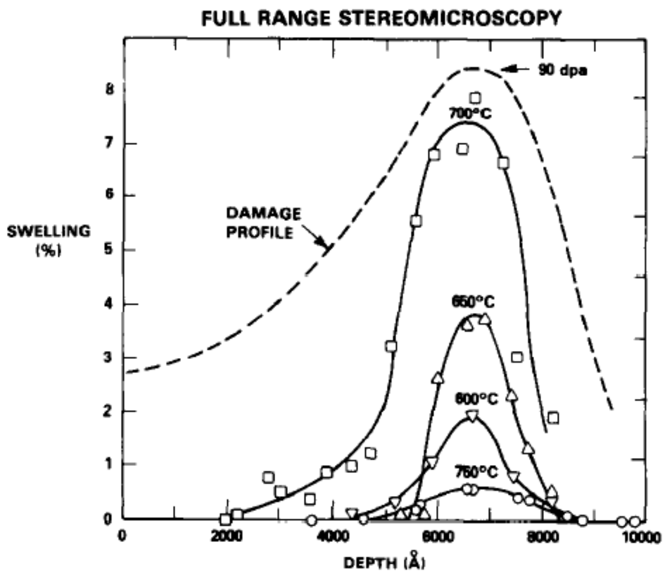

Fig. 1. Comparison of damage profile curve and swelling determined at 90 dpa by Diamond and coworkers for the ADIP Alloy [12].

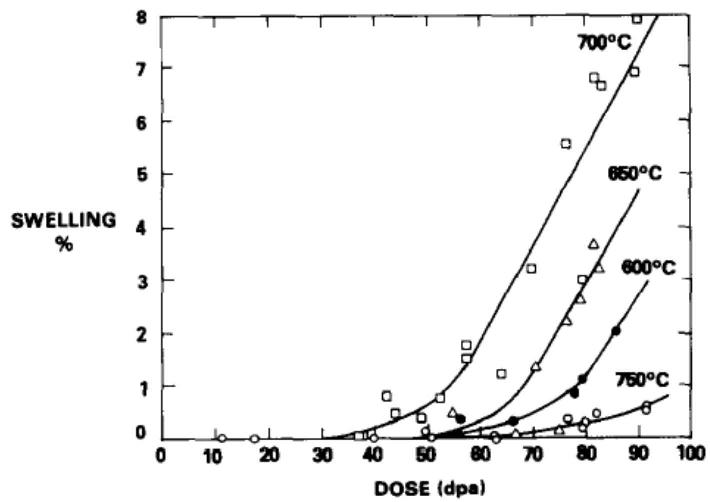

Fig. 2. Swelling versus dose profiles for the ADIP alloy, derived from fig. 1 [12].

Extraction of swelling data used HVEM over entire ion range, reducing scatter and depth uncertainties.

[12] S. Diamond, M.L. Bleiberg, I.M. Baron, R. Bajaj and R.W. Chickering, in: Radiation Effects and Tritium Technology for Fusion Reactors, CONF-750989 (Gatlinburg, TN, October 1–3, 1975) p. I-207.

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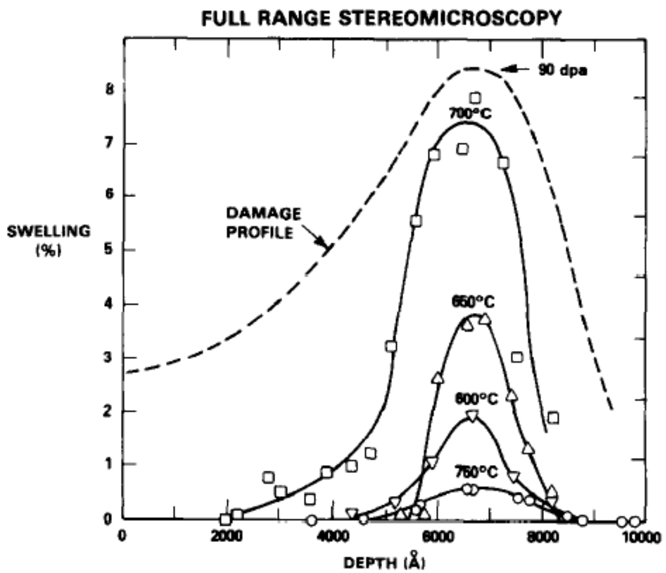

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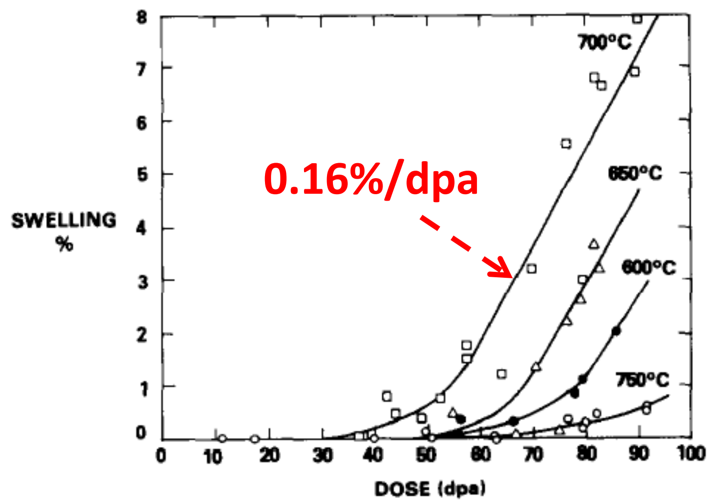
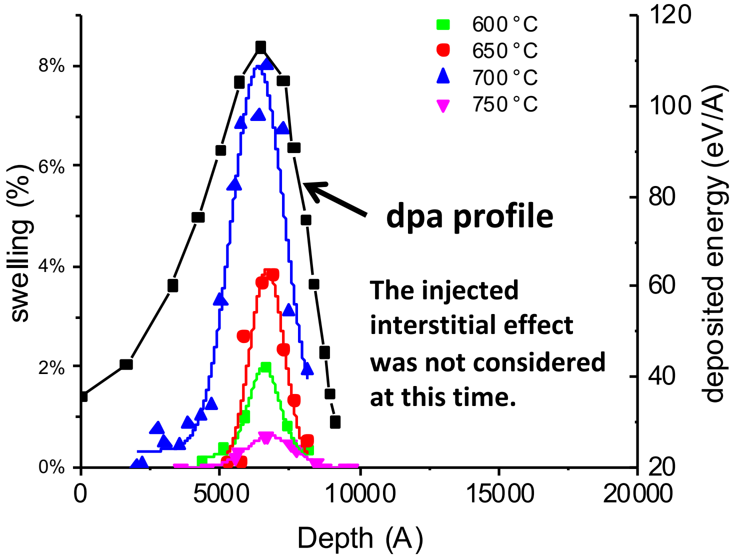


Fig. 2. Swelling versus dose profiles for the ADIP alloy, derived from fig. 1 [12].

Extraction of swelling data used HVEM over entire ion range, reducing scatter and depth uncertainties.

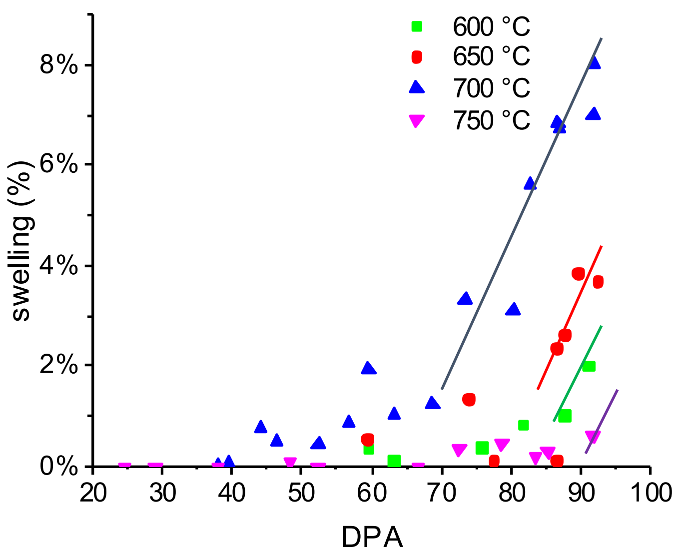
Original interpretation of 3.5 MeV Ni ion irradiation data on Fe-15Cr-25Ni



Peak damage dose of 90 dpa was calculated with EDEP to be ~700 nm.

Swelling peaks at peak dose position, confirming our expectation at that time.

Injected interstitial influence was either absent or was quickly overcome.

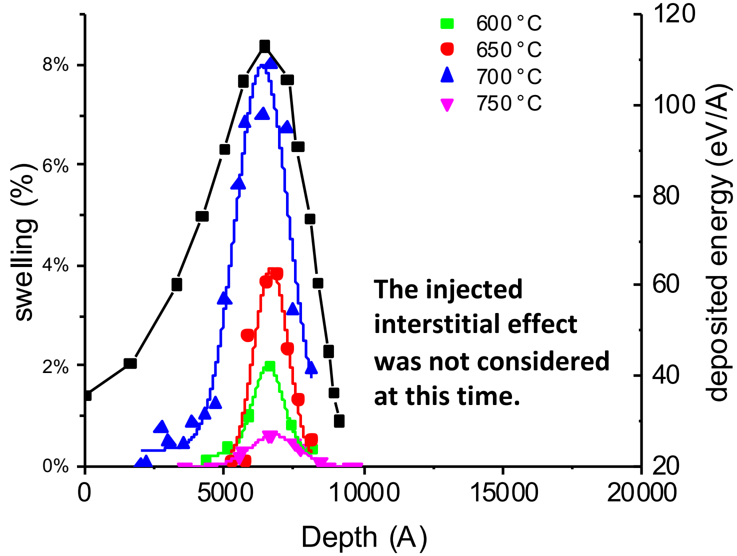


Plot swelling vs. local dpa level.

Swelling peaks at ~700°C due primarily to having the shortest incubation period.

Maximum post-transient swelling rate is on the order of ~0.20%/dpa, much lower than the ~1%/dpa observed in neutron irradiation.

Comparison: two interpretations of 3.5 MeV ion irradiation data on Fe-15Cr-25Ni



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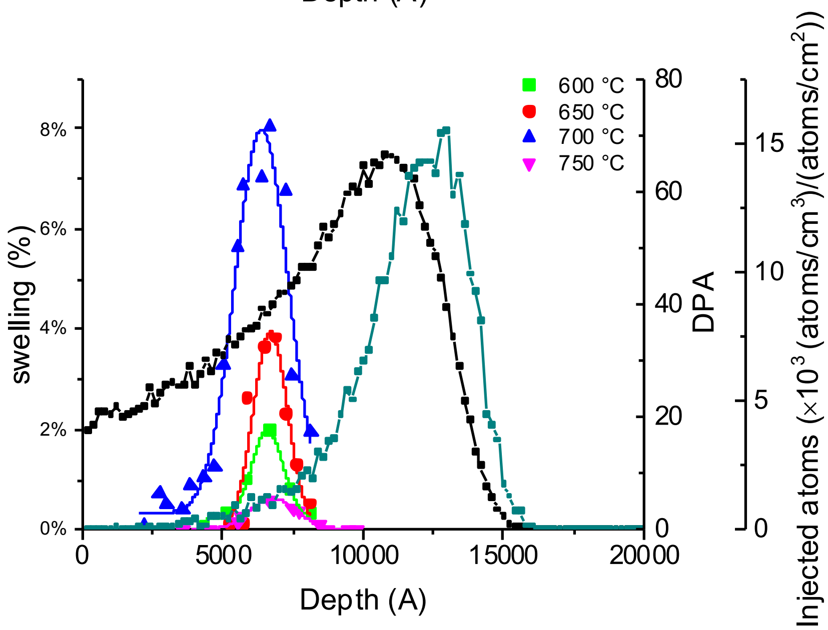
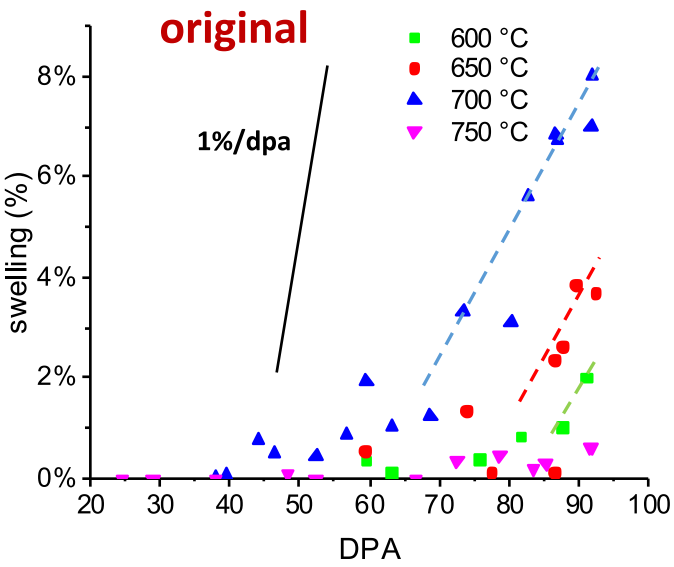


Peak damage dose was calculated with SRIM to be 67 dpa at ~1100nm.

The injected interstitial effect is very strong to suppress void nucleation in back half of ion range.

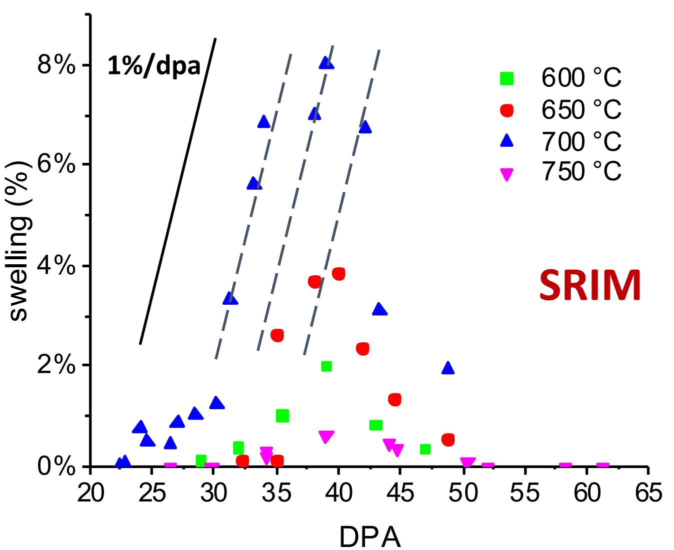
Assigned dpa levels drop strongly and post-transient swelling rates increase in region far from injected interstitial.

Comparison: two interpretations of 3.5 MeV ion irradiation data on Fe-15Cr-25Ni



Original interpretation was based on a dpa curve that was much shorter in range than would be calculated by SRIM.

One consequence is that the calculated dpa levels were too high.



Correct SRIM calculations yield much lower dpa levels and therefore higher swelling rates per dpa.

Data taken far from injected interstitial implies a post-transient swelling rate of ~1%/dpa, similar to that observed in neutron irradiation.

Charged particle irradiation of neutron-preconditioned 316 stainless steel

20% CW 316 irradiated in EBR-II to 46.5 dpa at 585°C followed by 4 MeV Ni ion irradiation

Lauritzen et al. 1979,

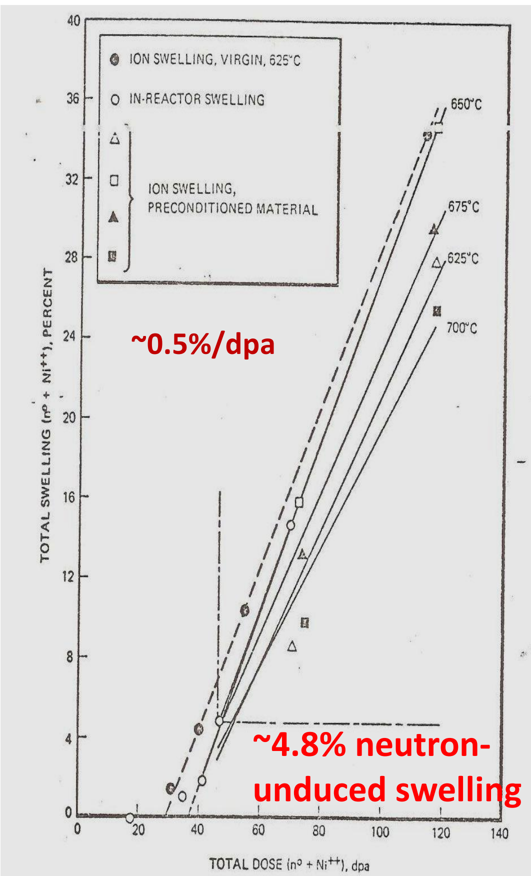


FIGURE 2. DOSE DEPENDENCE OF SWELLING IN AISI 316-STAINLESS STEEL. ALL DATA PLOTTED ON A LEAST SQUARES LINEAR FIT. CONVERSION OF NEUTRON DOSE TO dpa WAS BASED ON THE RELATION 10^{23} n/cm^2 ≡ 50 dpa

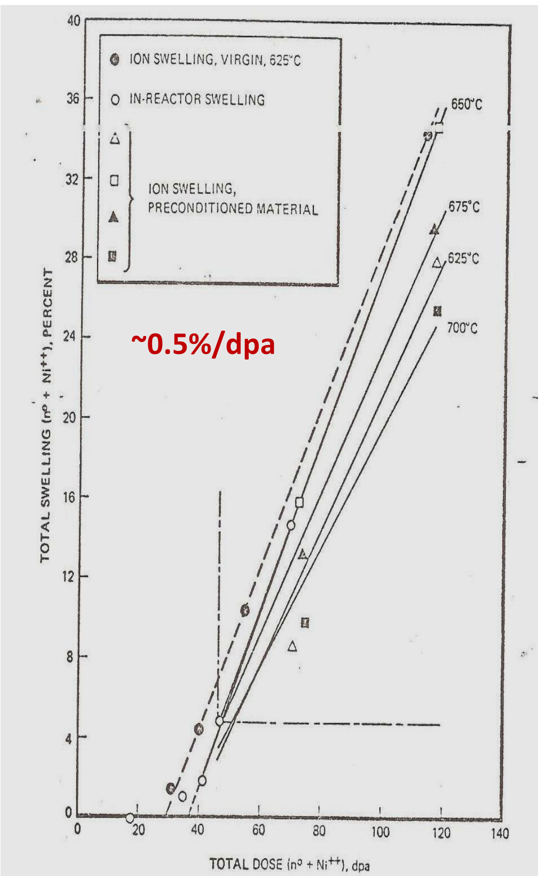
Note that the virgin specimen was irradiated with 5 MeV Ni ions at 6 x 10^{-3} dpa/sec, while the EBR-II specimens were irradiated at 2.4 x 10^{-2} dpa/sec.

Believing in a temperature shift, ion irradiation proceeded at four temperatures 40 to 115°C higher than the original neutron irradiation.

Charged particle irradiation of neutron-preconditioned 316 stainless steel

20% CW 316 irradiated in EBR-II to 46.5 dpa at 585°C followed by 4 MeV Ni ion irradiation

Lauritzen et al. 1979, modified by Garner 2016



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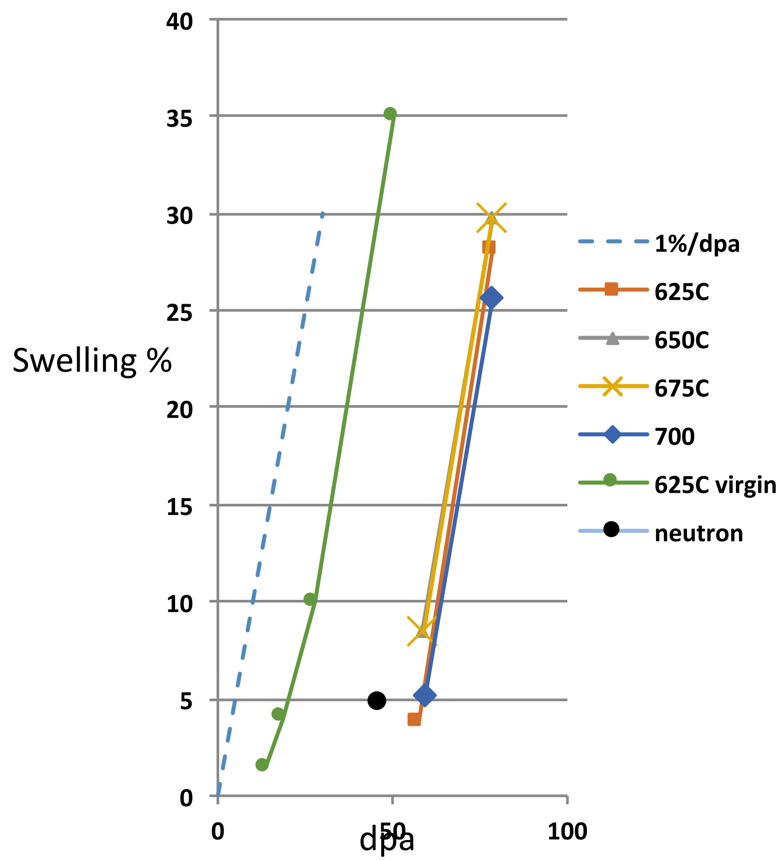

Note that the virgin specimen was irradiated with 5 MeV Ni ions at 6 x 10^{-3} dpa/sec, while the EBR-II specimens were irradiated at 2.4 x 10^{-2} dpa/sec.

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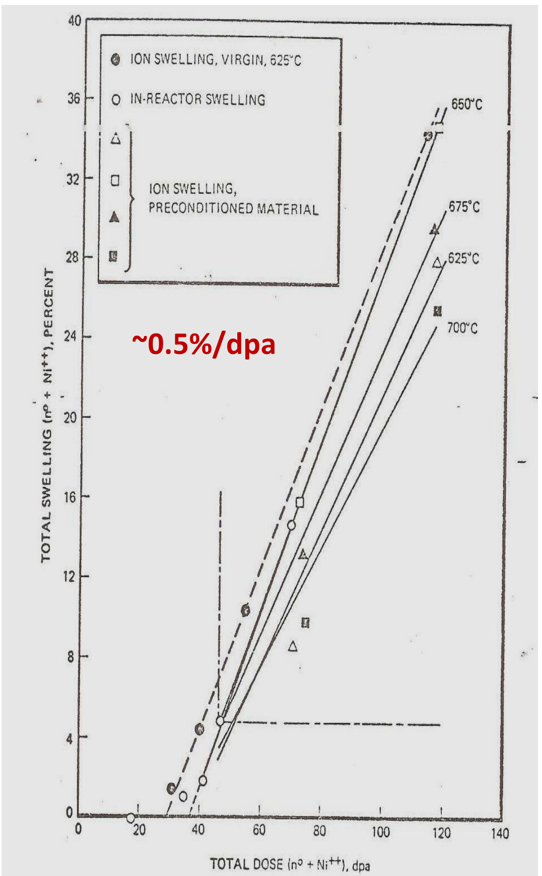

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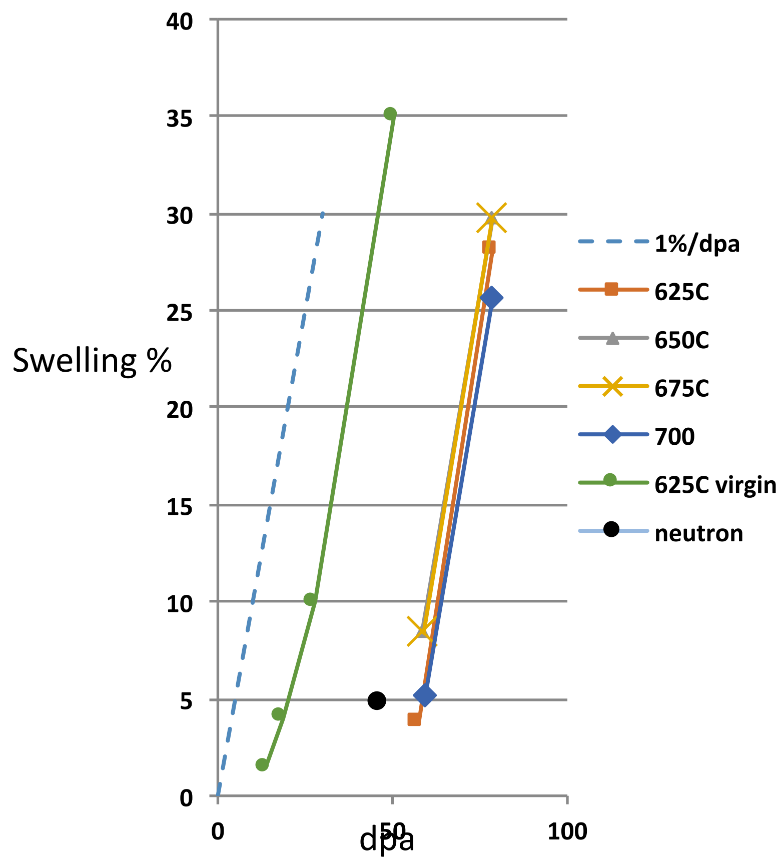


Note that the virgin specimen was irradiated with 5 MeV Ni ions at 6 x 10^{-3} dpa/sec, while the EBR-II specimens were irradiated at 2.4 x 10^{-2} dpa/sec.

The irradiation temperature showed no influence.

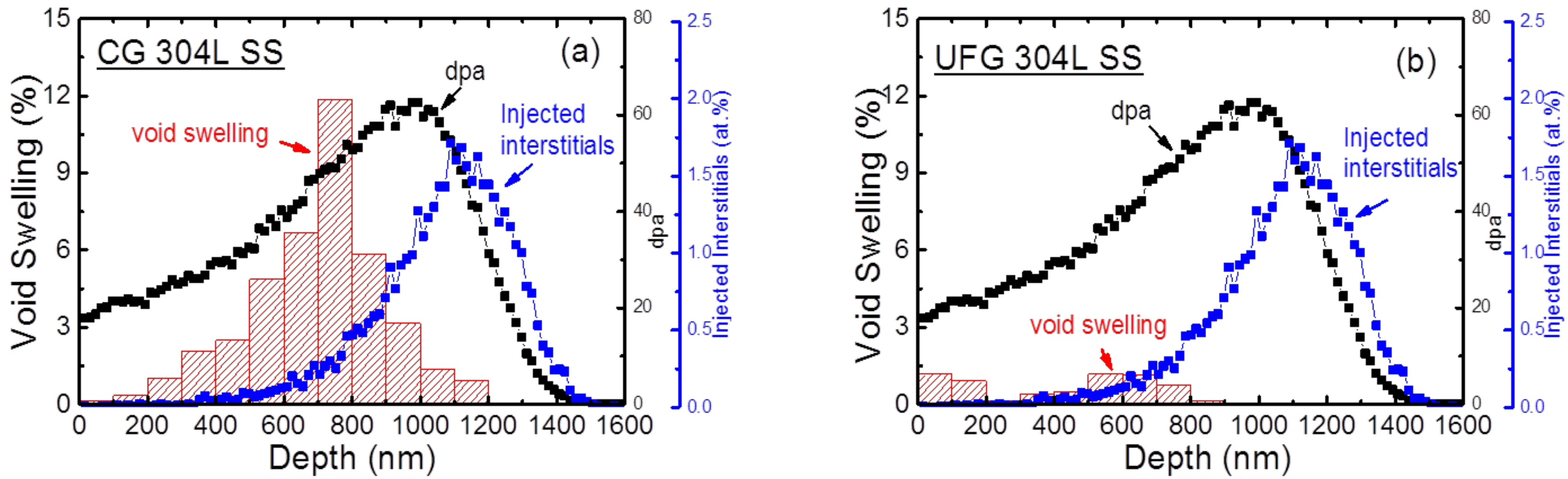
The temperature shift did not affect the swelling rate.

It appears that use of more modern energy loss parameters in SRIM significantly improves the credibility of earlier experiments.

- **Other earlier-generation studies not shown in this presentation also confirm the improved credibility of revised interpretations based on SRIM-calculated energy deposition values.**
- **Let’s look at a recently conducted study on austenitic 304 stainless steel.**
- **Will we see the same features of injected interstitial suppression, remnant swelling peaks and neutron-typical post-transient swelling rate?**

3.5 MeV Fe irradiation of two variants of 304L stainless steel to 60 peak dpa at 500°C (submitted to NIMB, 2016)

C. Sun, F. A. Garner, Jing Wang, L. Shao, X. Zhang, S. A. Maloy



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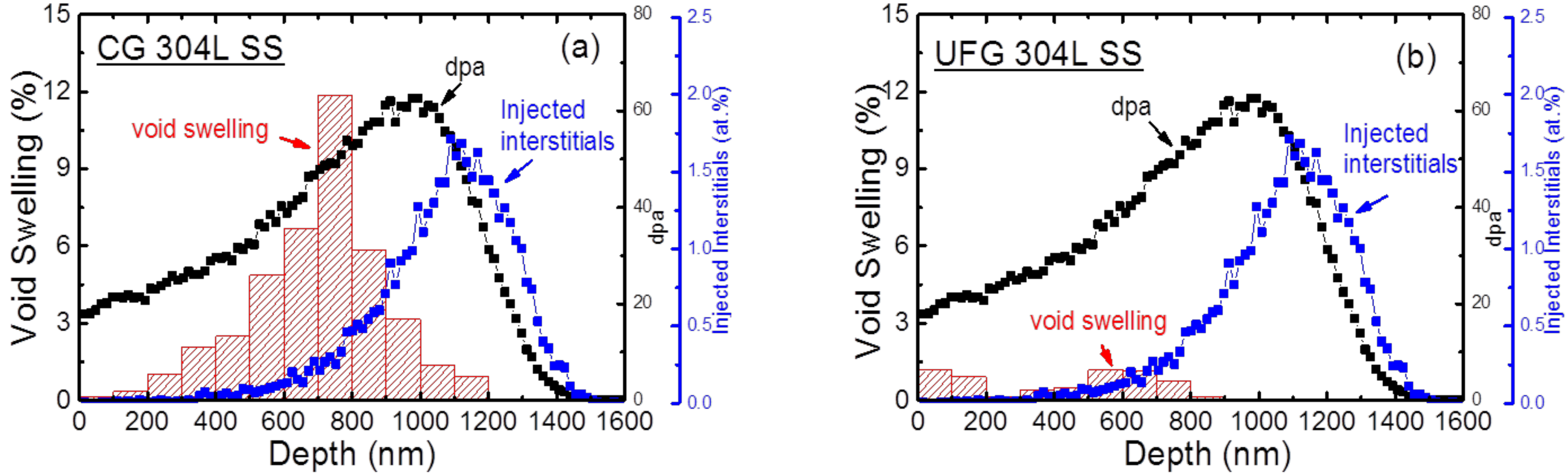
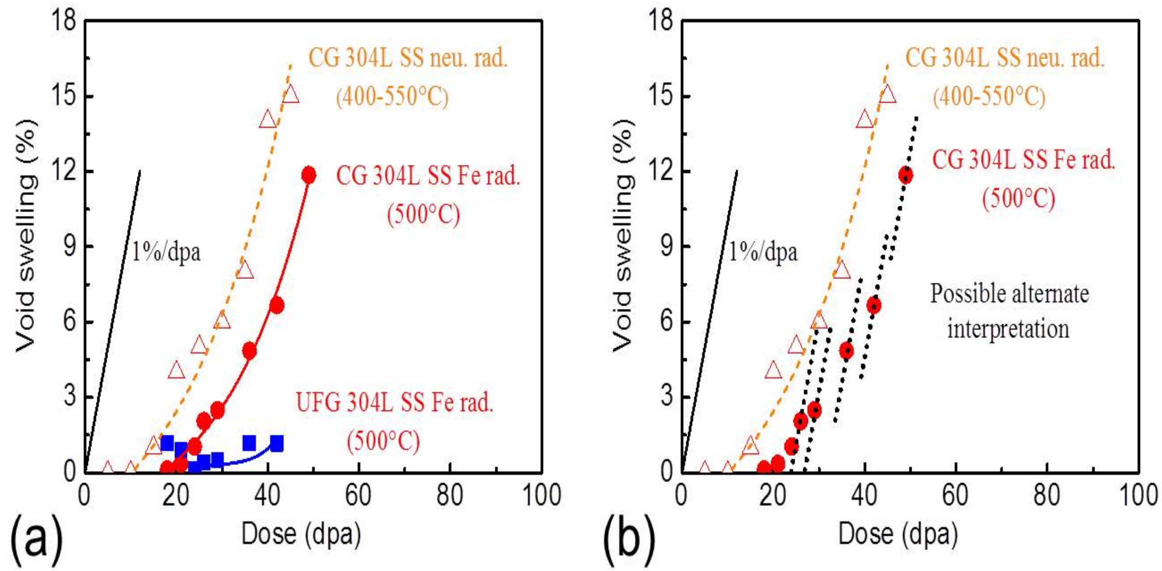




Conclusions

- **I have shown only a fraction of the earlier work, but the overall effort yields very satisfying results when reevaluated using modern energy deposition values in SRIM.**
- **The net result of this reevaluation is that studies conducted much earlier were only deficient in the values of dpa calculated for each swelling datum.**
- **It now appears that self-ion bombardment can predict the steady-state swelling rates of both fcc and bcc iron-base alloys.**
- **There is some additional support for the effect of increasing dpa rate to extend the transient regime of swelling.**
- **The injected interstitial suppression of void nucleation is <u>equally</u> powerful in both fcc and bcc iron-base alloys.**

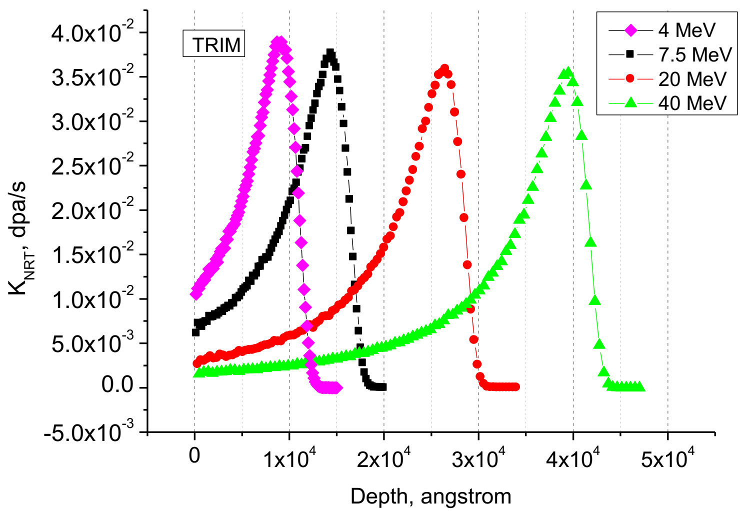
- **Bottom line: ion simulation of neutron-induced void swelling is more credible than previously realized.**

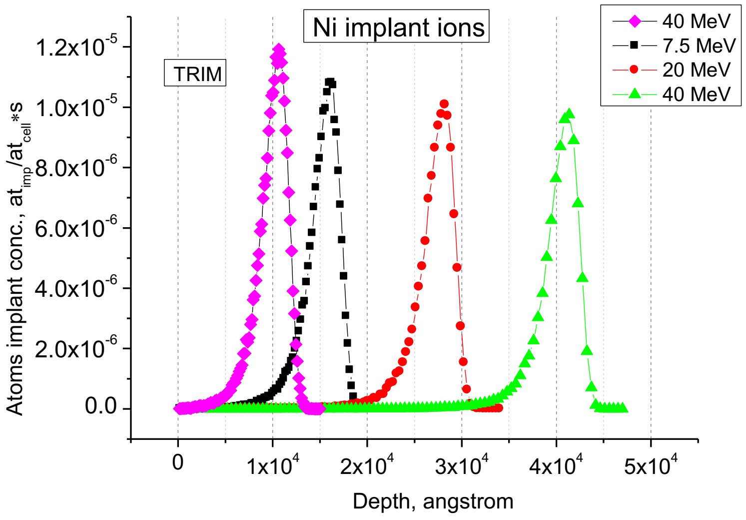
Some cautions

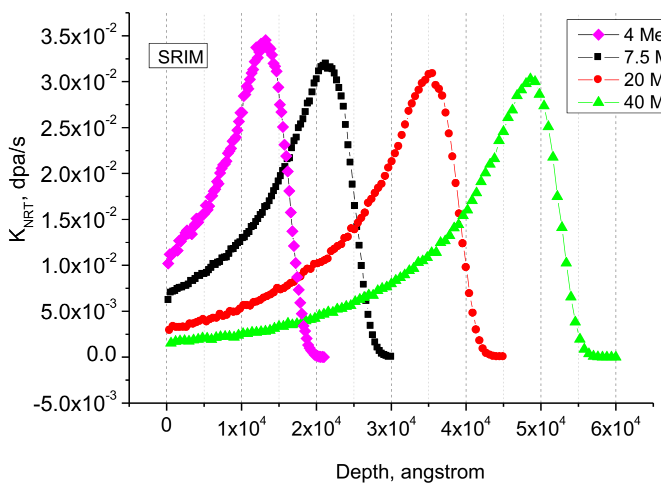
- **In comparing older data with your new results, most older swelling data points will require reevaluation of the dpa values.**
- **Additionally, many of the earlier experiments used displacement threshold energies of 25 or 33 eV, requiring further modification to allow comparisons between older and newer experiments.**
- **There are still smaller but significant differences between dpa curves produced by various generations of TRIM and SRIM, requiring additional caution in comparison.**

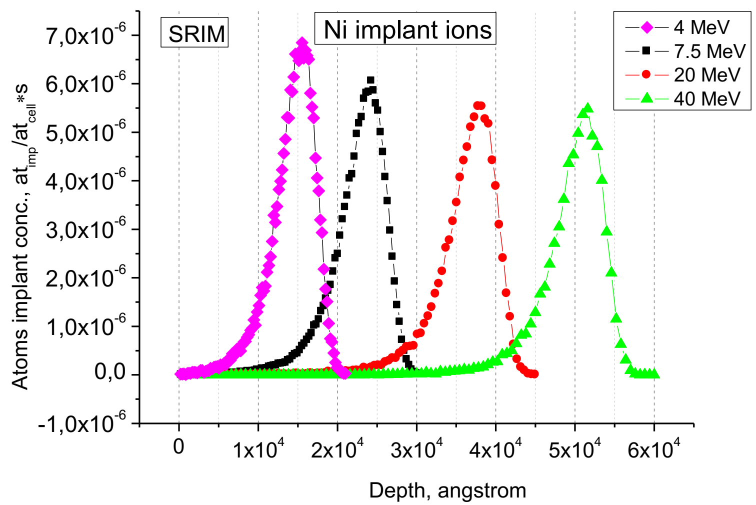
Back-up slides

Input from Valery Pechenkin on comparison of TRIM-98 and SRIM-2012

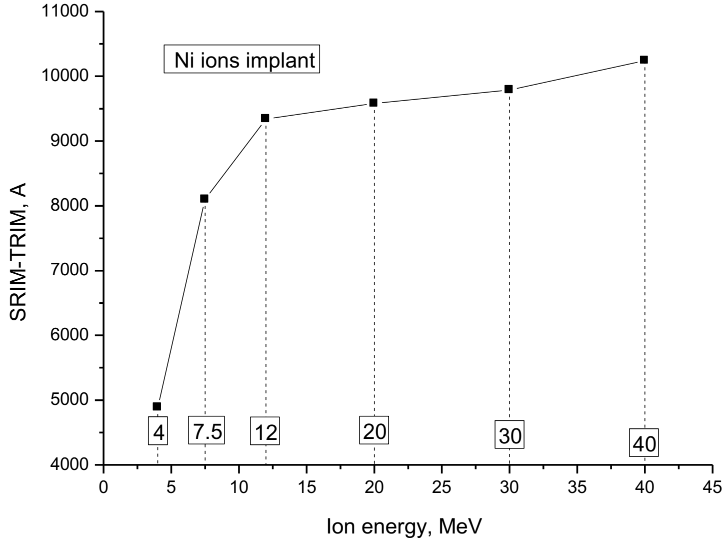




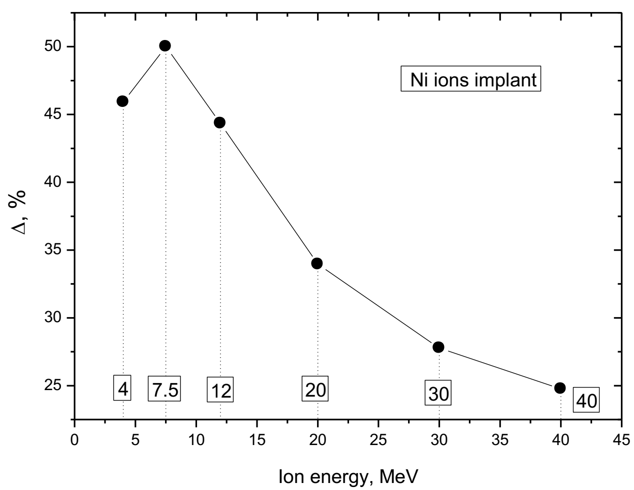




Input from Valery Pechenkin of IPPE, Russia



$$\Delta = \frac{SRIM - TRIM}{TRIM} \cdot 100\%$$

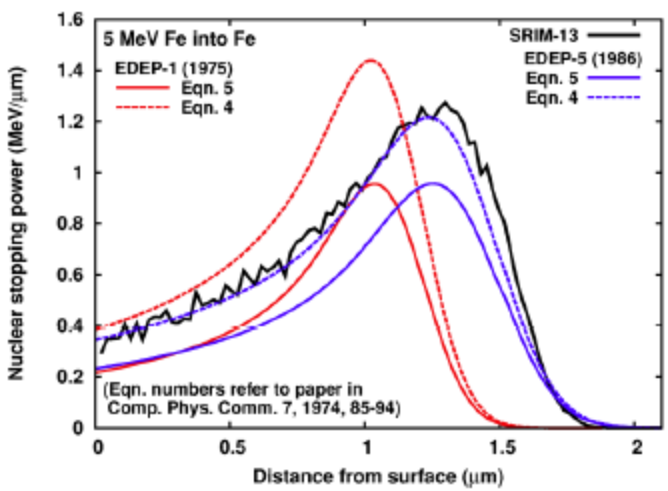


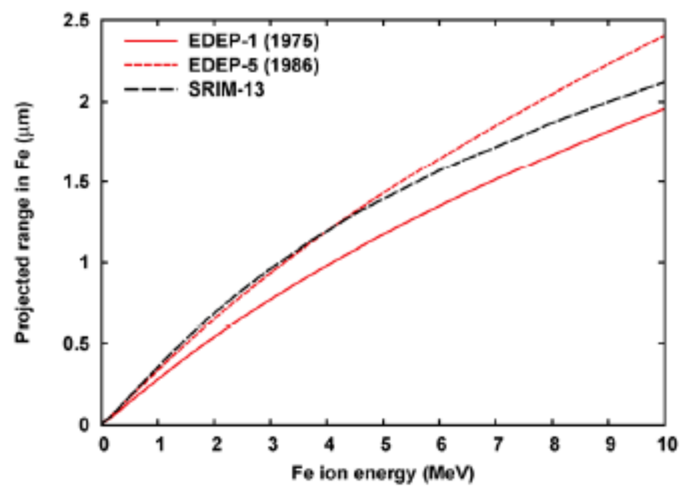
Difference between locations of Ni ion implantation peaks

Calculations with TRIM-98 and SRIM-2012 of Ni ion implantation (4 - 40 MeV) in EP-450.

The difference increases with increasing ion energy in the range of our interest. The difference eventually tends to saturate , but only at energies above our interest.

Input from Roger Stoller

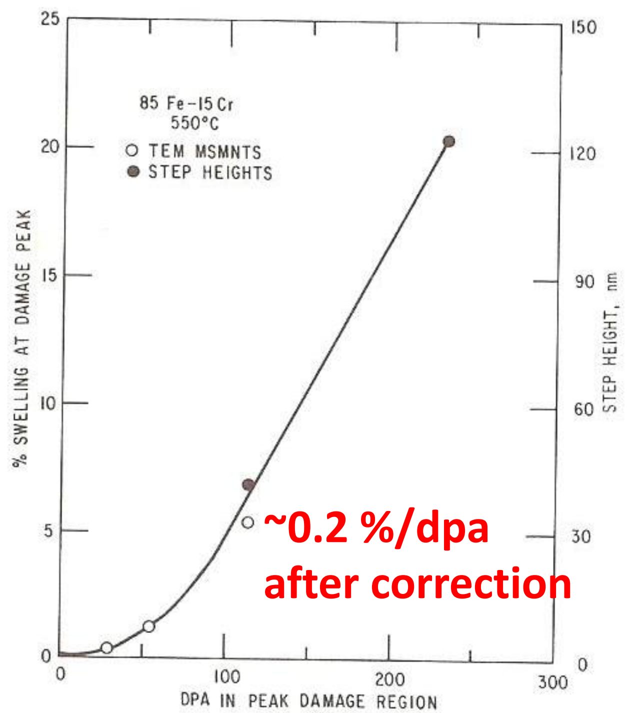




For your entertainment and perhaps interest, after your seminar I was able to obtain two versions of EDEP on line. One is the original version from Manning and Mueller's 1974 paper with some corrections made in 1975, and the other is a 1986 update with what they called better stopping powers. Surprisingly, it was not too bad of a job to get these old codes to compile and run so I could make a simple comparison with SRIM-2013 for Fe into Fe. The results are shown on the next page. For Fe ion energies up to about 6 MeV EDEP-5 is pretty consistent with SRIM-2013, above that energy SRIM falls between EDEP-1 and EDEP-5.

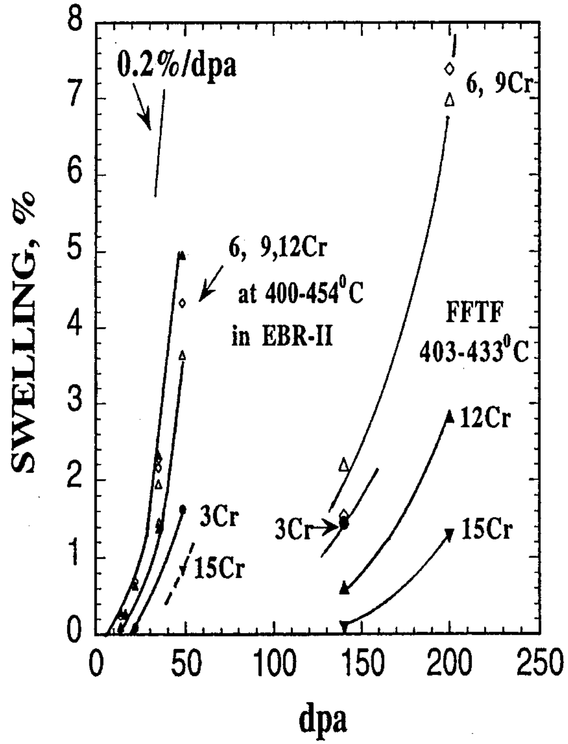
Comparison of ion and neutron results on bcc Fe-Cr binary alloys

5 MeV Ni^+ ions



Johnston et al.

EBR-II and FFTF



Garner, Toloczko and Sencer

Both techniques show <u>bilinear</u> swelling behavior with ~0.2%/dpa steady-state swelling rate for bcc iron alloys.