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Microstructure, precipitate and property evolution in cold-rolled Ti-V high strength low alloy steel

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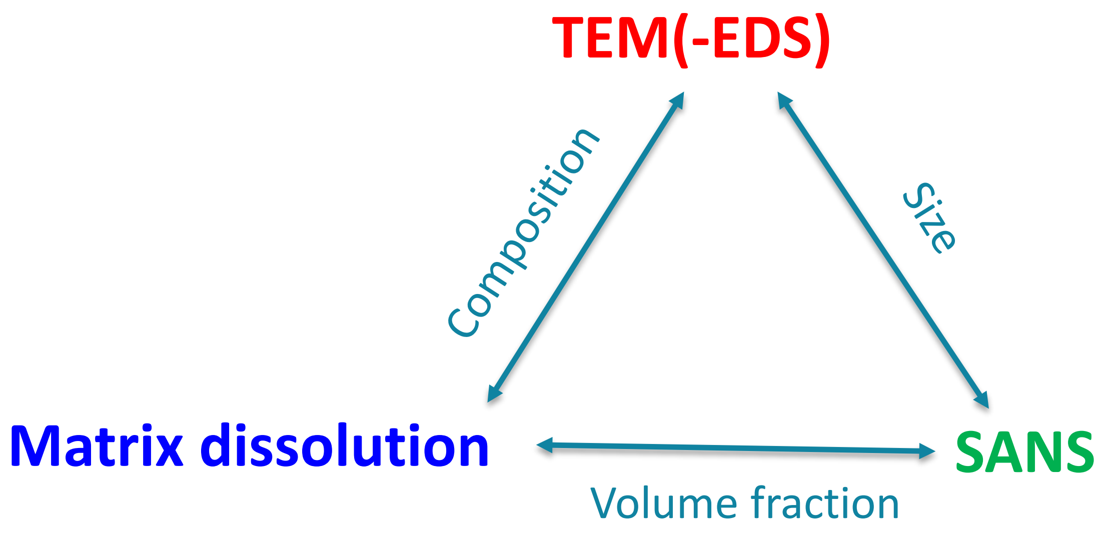
1. Introduction

- High strength low alloy (HSLA) steels are applied in automotive industry.
- Cold-rolled and annealed (vs hot rolled): lower strength, higher elongation.
- Cold rolled Ti-V HSLA steels are promising for better performance and low costs.
- Nanoscale precipitates play an important role, but received little attention.

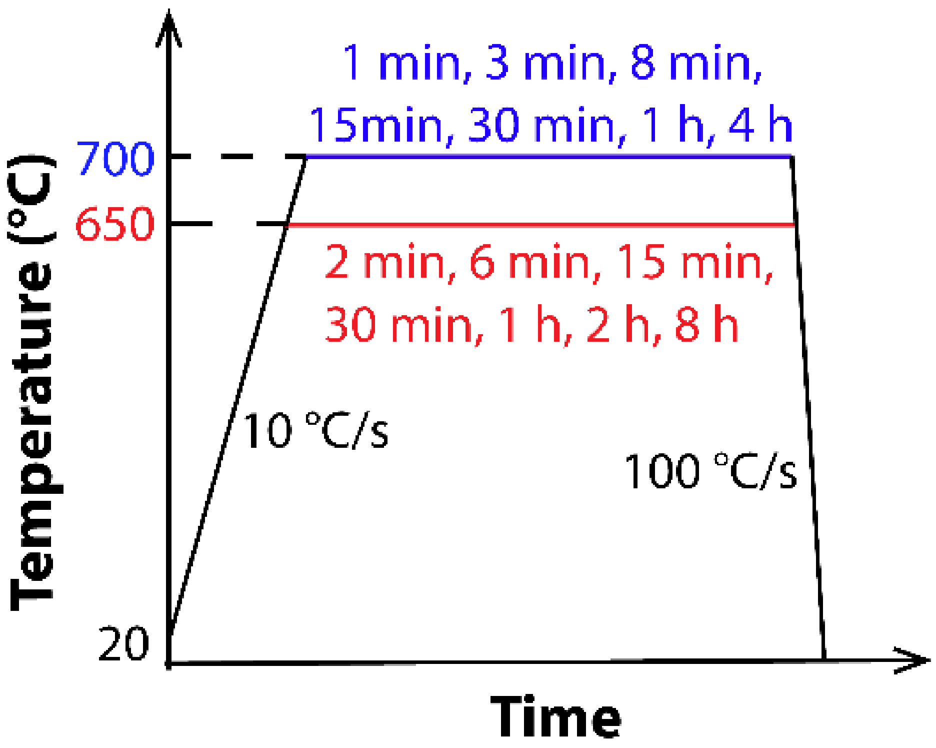
1. Aim

- Providing in-depth understanding of precipitation behavior, microstructure and mechanical property evolution in a cold rolled Ti-V HSLA steel;
- Testing and comparing accurate quantification methods of precipitate composition, size and volume fraction.

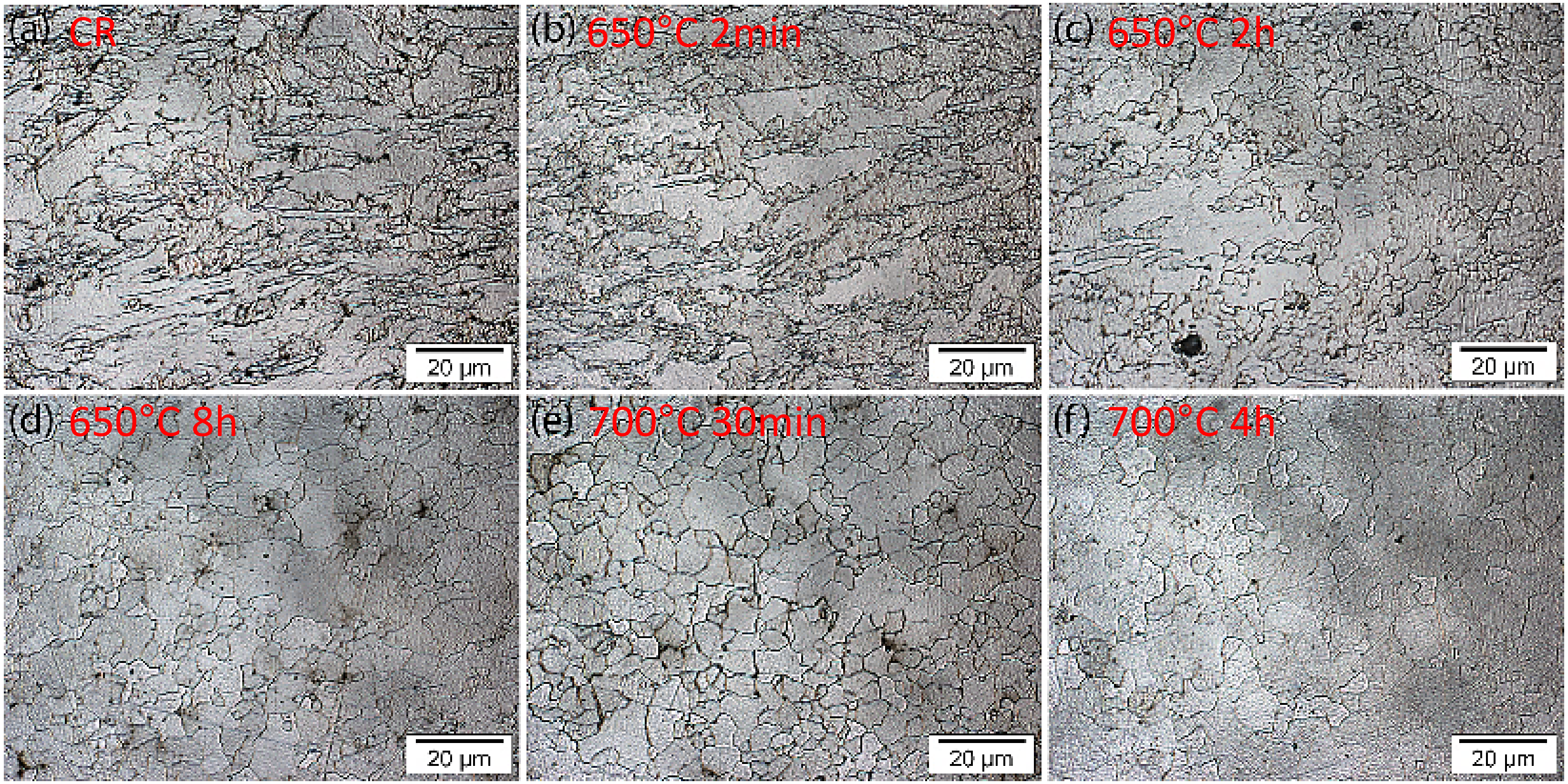


2.1 Sample preparation

	C	Mn	N	Ti	V
wt%	0.03-0.07	0.8-1.2	0.002-0.006	0.03-0.08	0.05-0.12

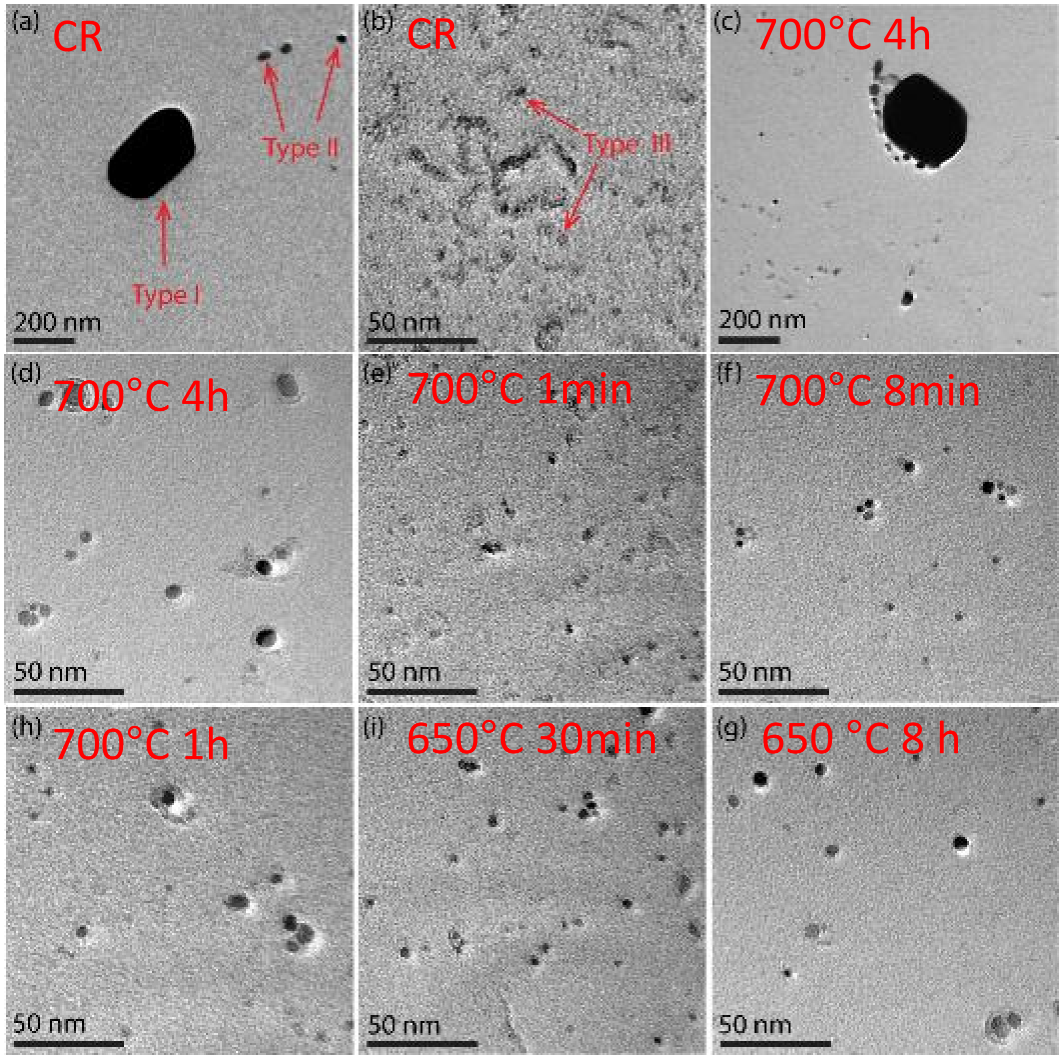


2.2 Light Microscopy



Recrystallization is (nearly) complete after 8 h at 650 °C and 30 min at 700 °C.

2.3 TEM (on carbon replicas)



Three types of precipitates:

Type I: 150- 500 nm, prismatic;

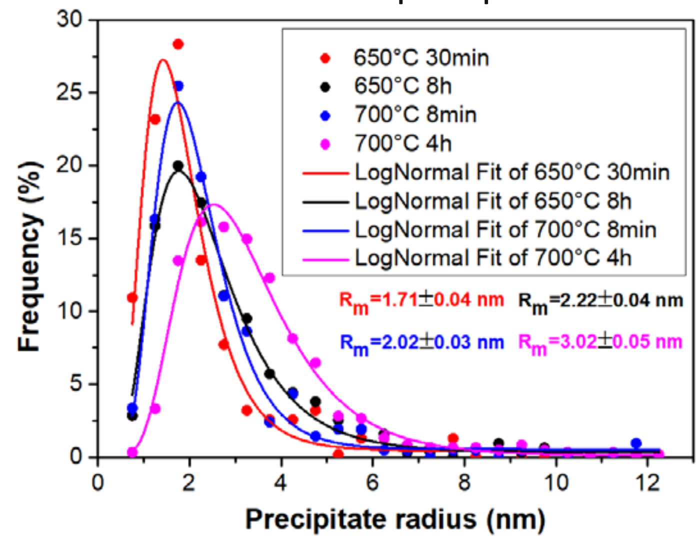
Type II: 30-70 nm, ellipsoidal;

Type III: 1- 30 nm, spherical.

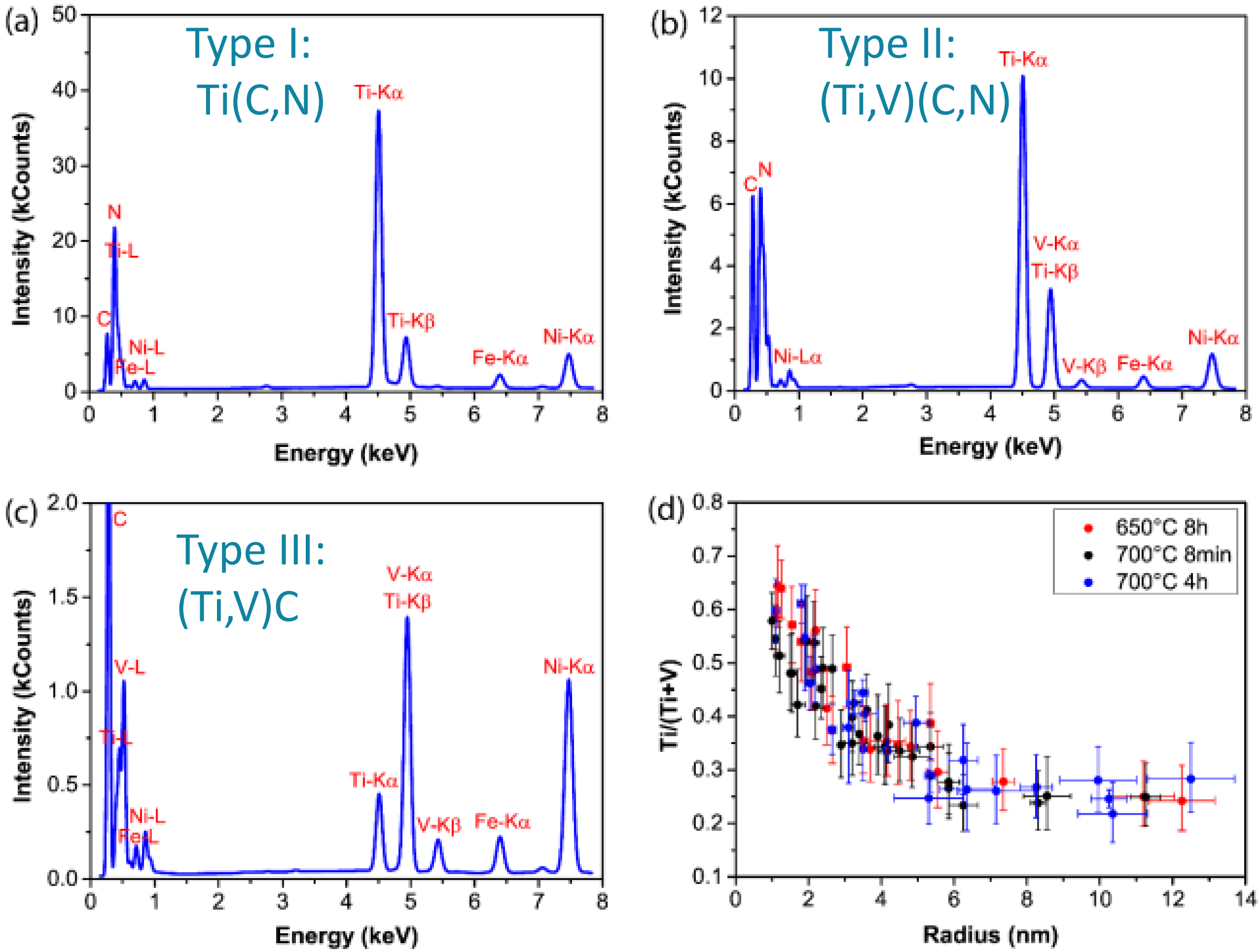
Type I and II: stable;

Type III: grow.

300~500 precipitates

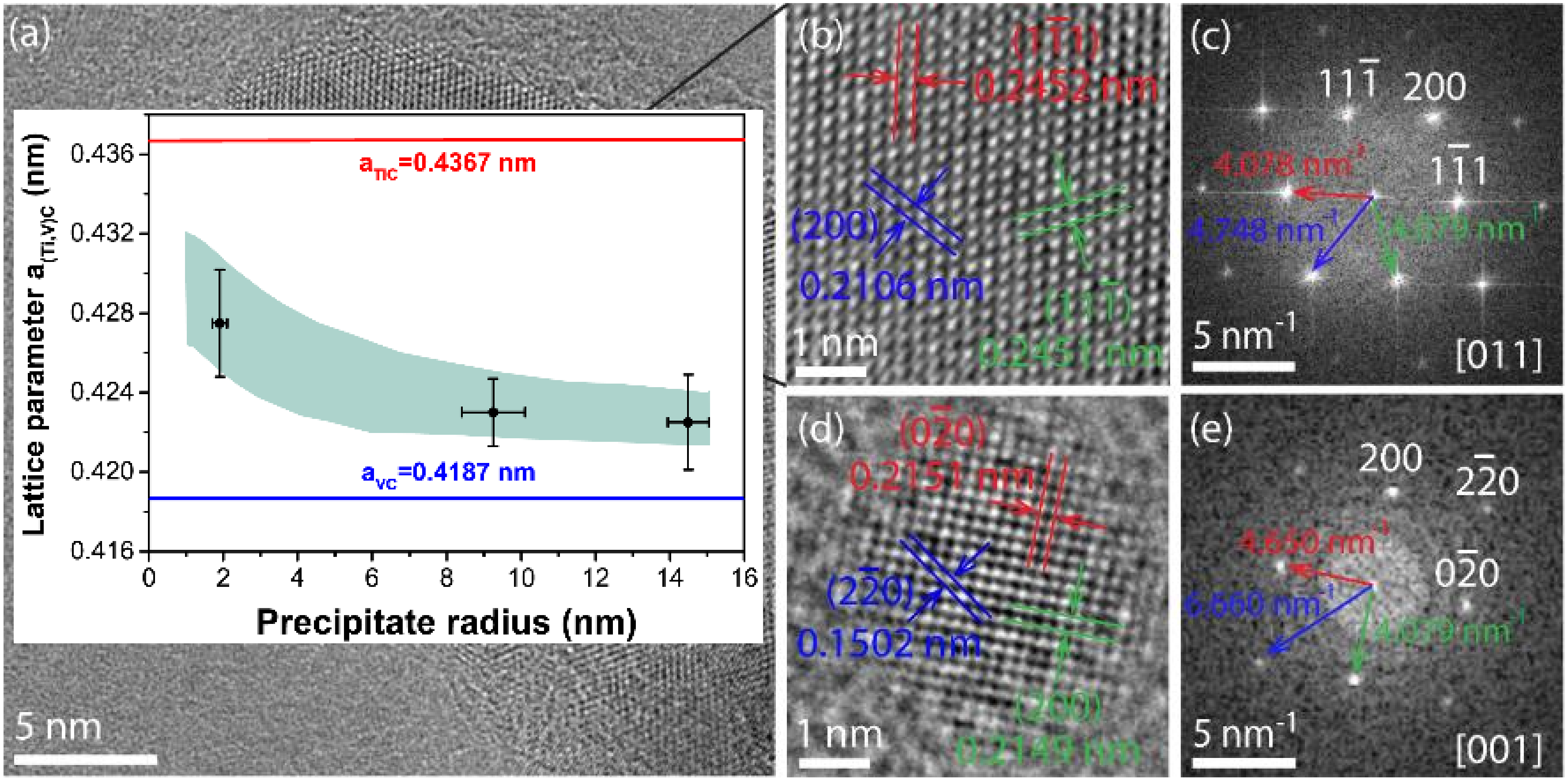


2.3 TEM-EDS



Type III: Ti/(Ti+V) atomic ratio decreases with increasing precipitate size. The core is Ti-rich.

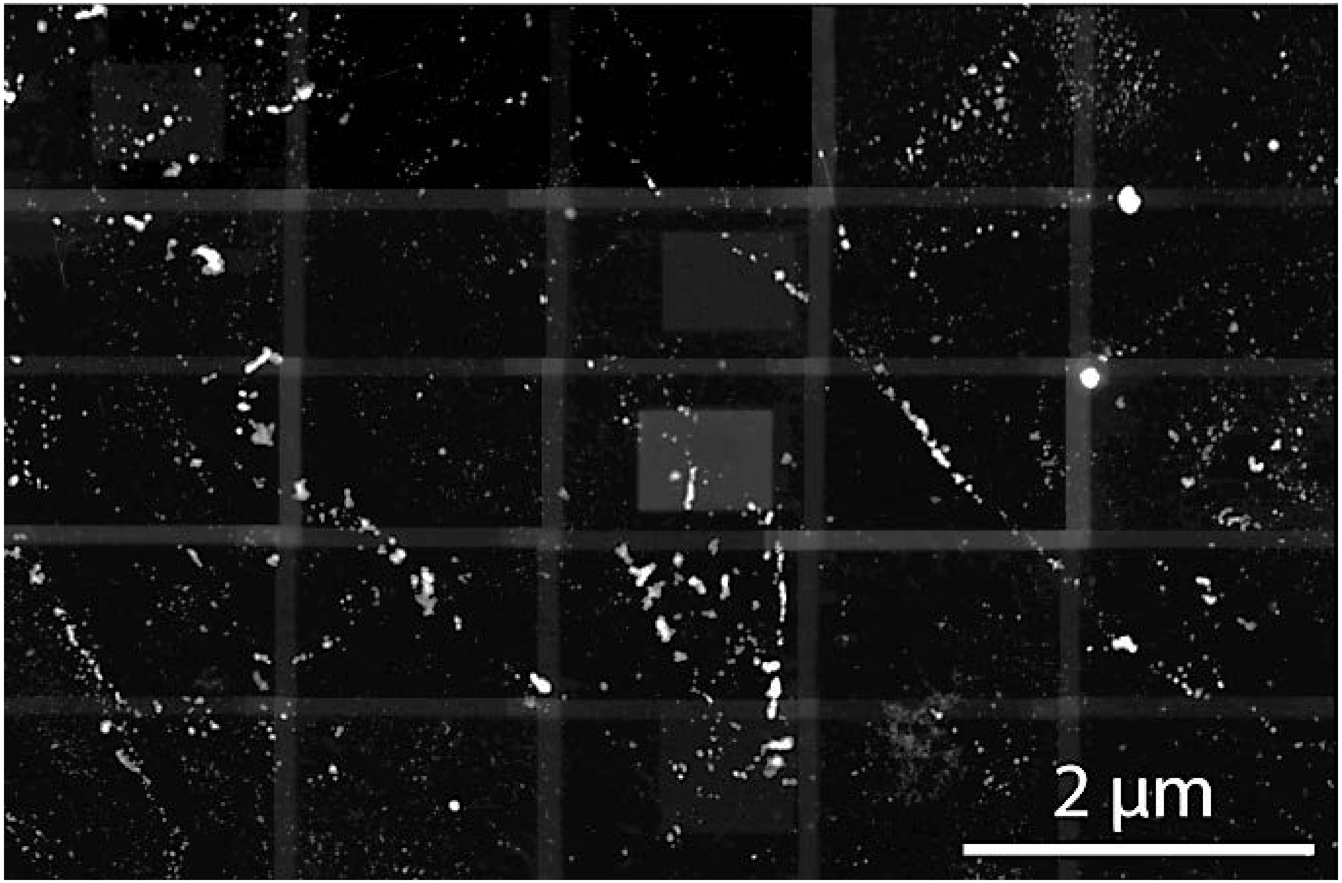
2.3 HRTEM



$$a_{(Ti_xV_{1-x})C} = x \cdot a_{TiC} + (1-x) \cdot a_{VC}$$

(Ti,V)C lattice parameter decreases with increasing precipitate radius.

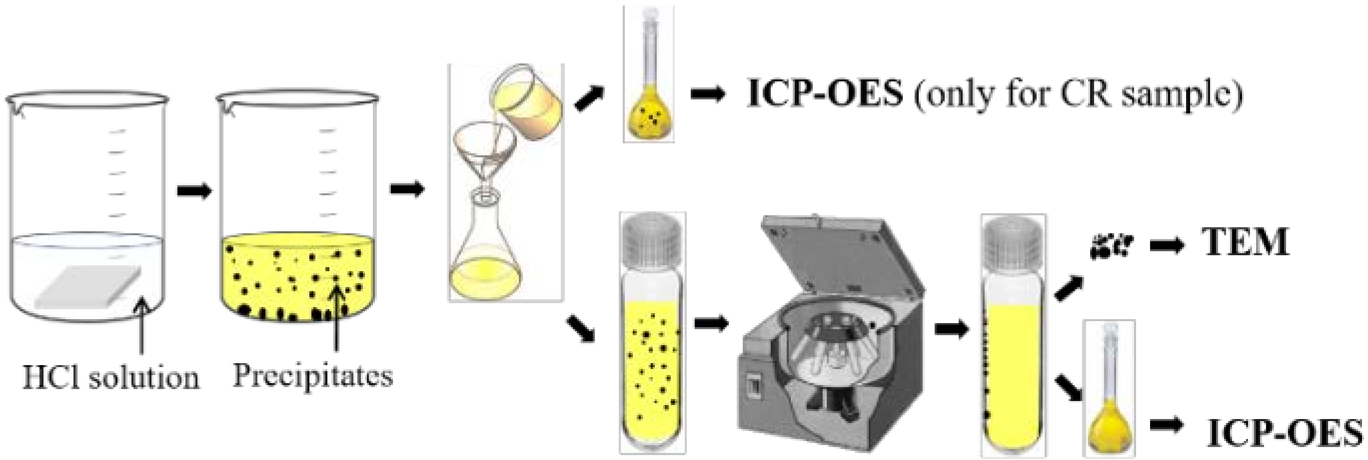
2.3 HAADF-STEM (in SEM) stitched image



- Type III (Ti,V)C precipitates are non-uniformly distributed.
- Bigger precipitates on grain boundaries; smaller ones on dislocations.

2.4 Matrix dissolution

Chemical dissolution + Filtering (20 nm) + Centrifuging + ICP-OES



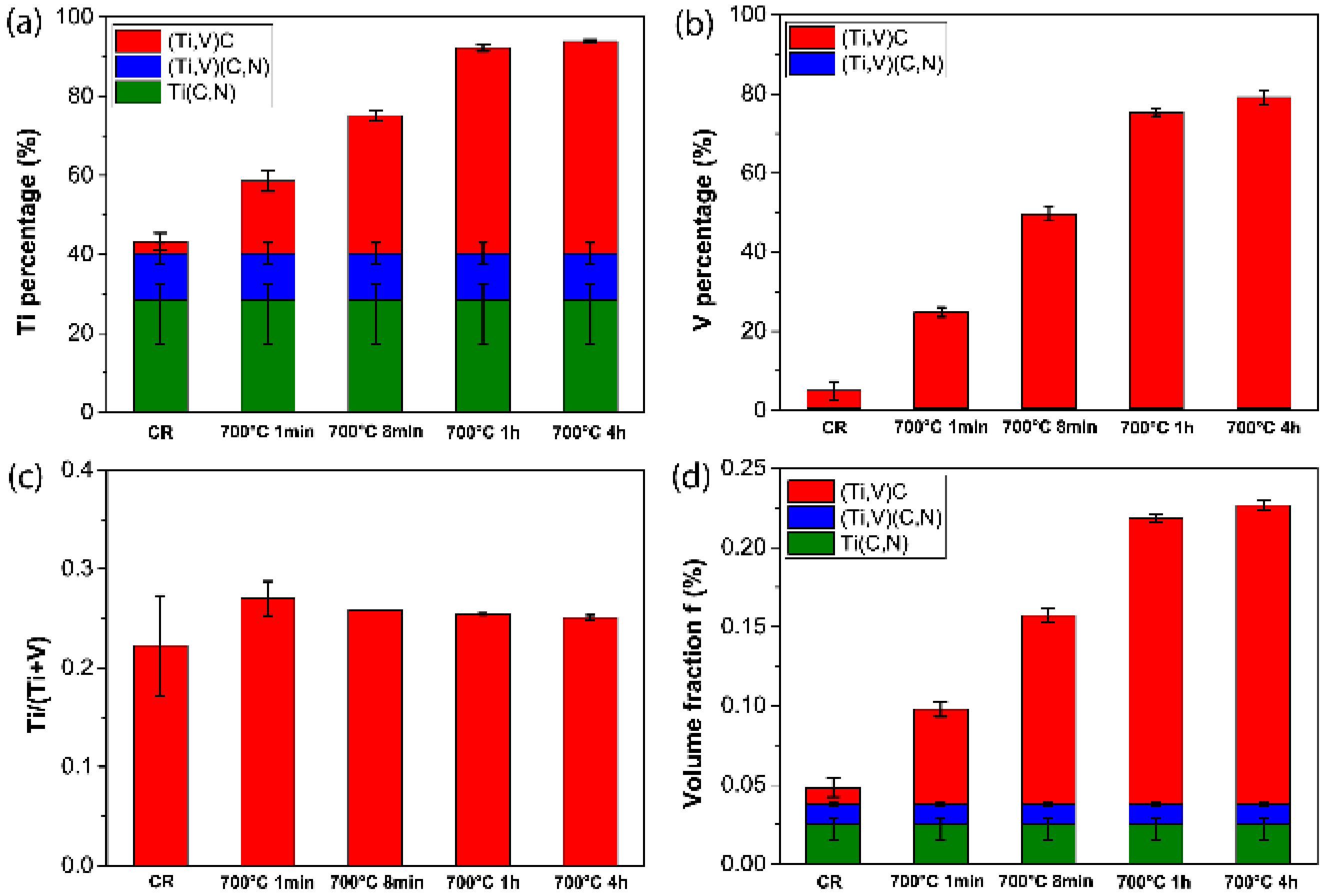
- **TEM:** Checking whether small precipitates have been centrifuged.
- **ICP-OES :** Obtaining Ti and V concentrations to calculate precipitate volume fraction.

  CR filtered: in type I & II precipitates;

  Centrifuged: in type III precipitates.

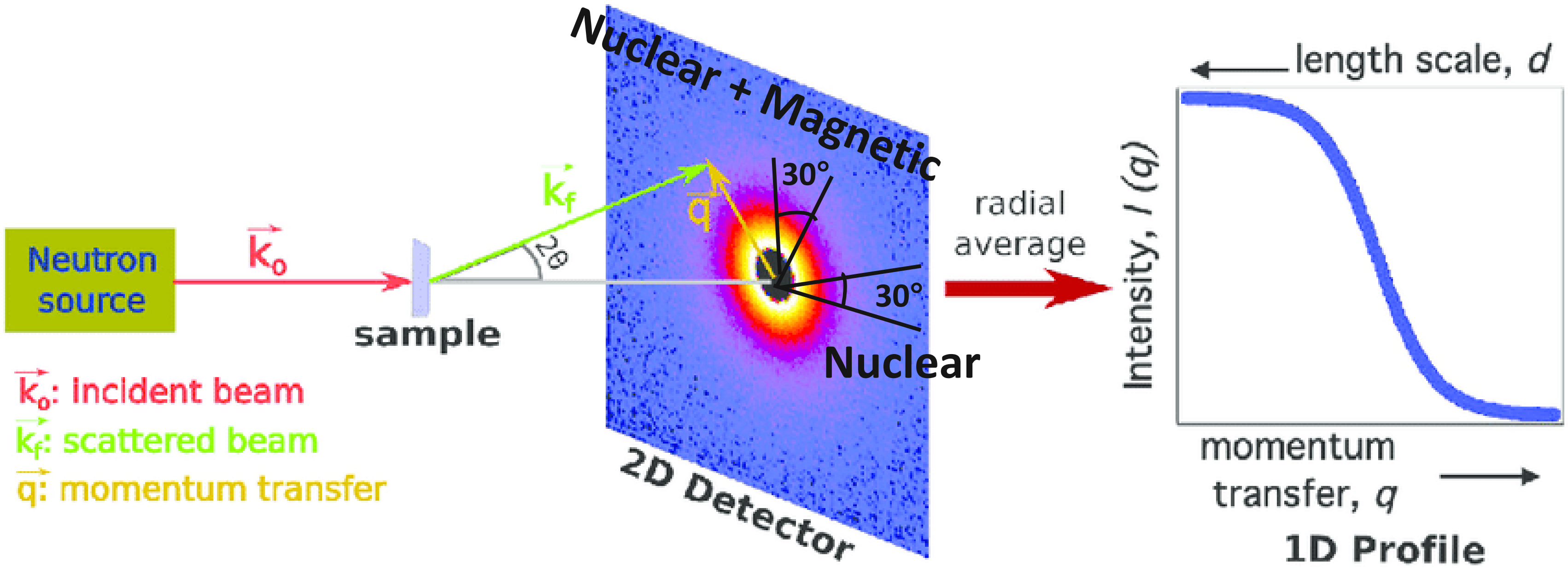
- Centrifuging efficiency is high.
- Precipitate dissolution is limited.

2.4 Matrix dissolution results



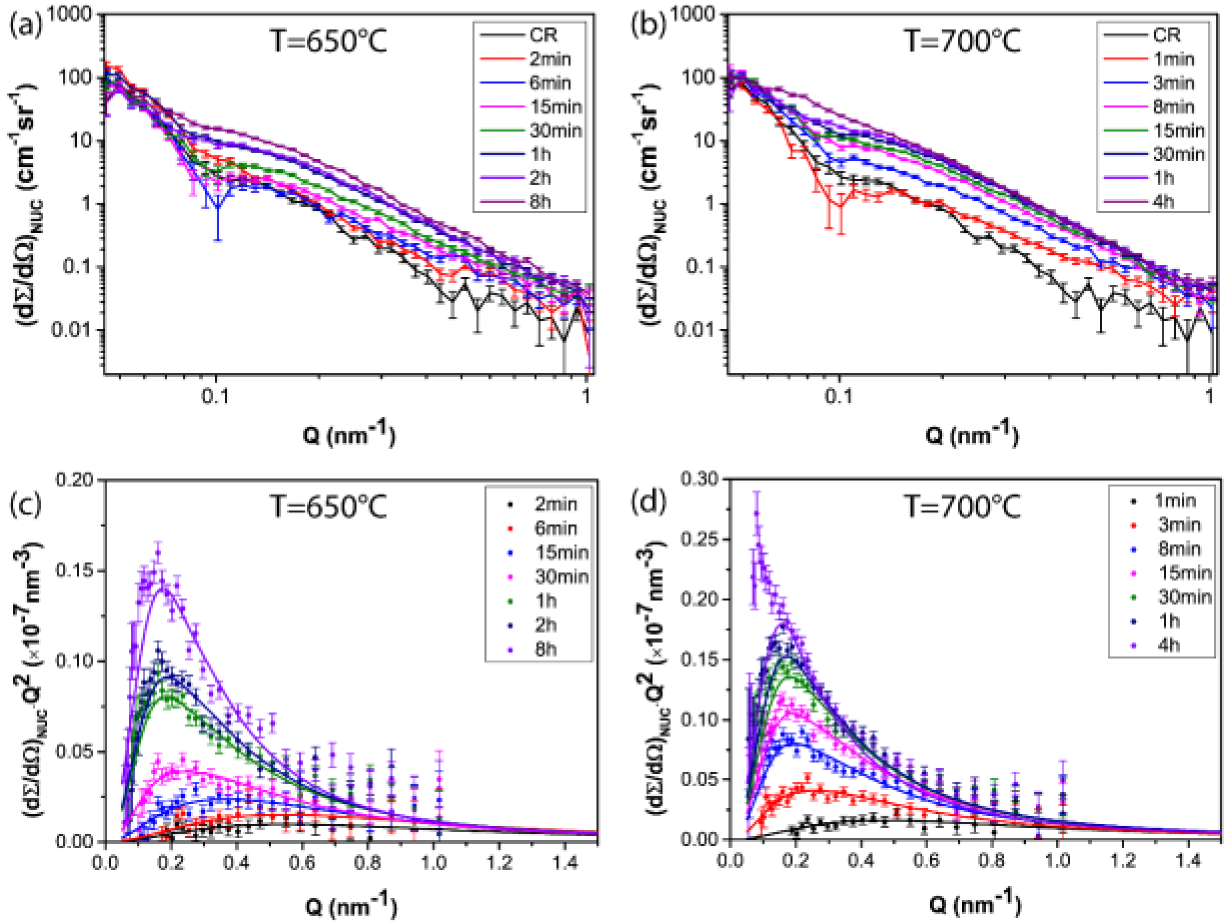
- Ti is dominant in (Ti,V)C and (Ti,V)(C,N), whereas V is dominant in (Ti,V)C.
- Separate volume fractions of the three types of precipitates were obtained; volume fraction of small precipitates is increasing.

2.5 SANS



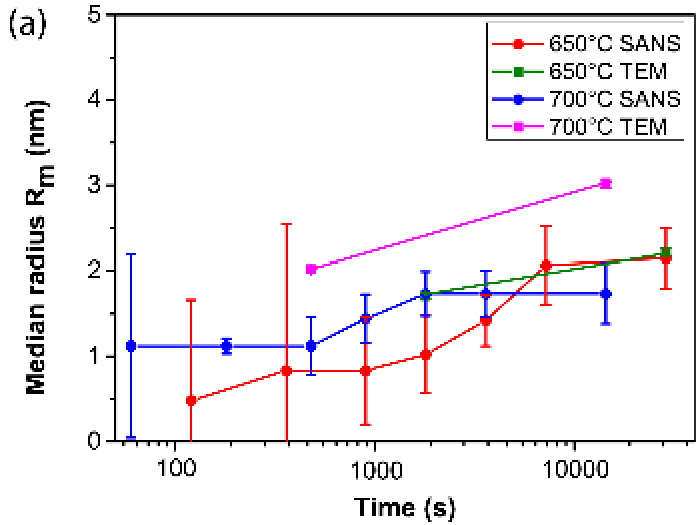
Nuclear scattering data was used for type III (Ti,V)C precipitate analysis after background subtraction of the CR sample.

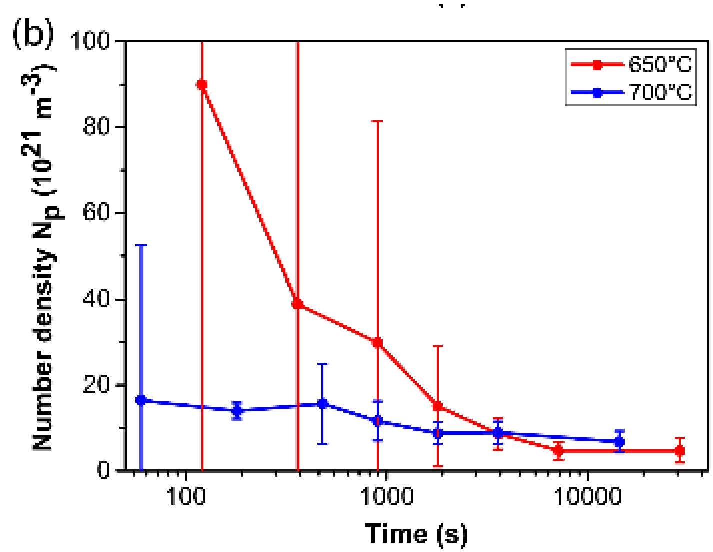
2.5 SANS

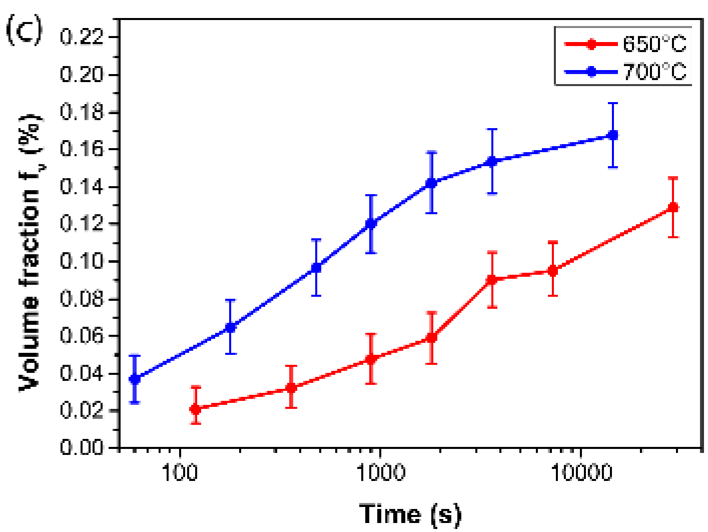


- SANS signal decrease is mainly caused by dislocation disappearance and sub-grain growth.
- Peak shifts towards smaller Q value, indicating the presence of larger precipitates.
- The poor Kratky plot fits for longer annealed samples are due to bigger precipitates on grain boundaries.

2.5 SANS results







- Preciptate median size increases (under-estimated for longer annealed samples). TEM result is generally higher than SANS result.
- Precipitate number density decreases; nucleation cannot be observed.
- Precipitate volume fraction increases.

2.6 Precipitate size comparison

- **TEM**: Median radius is overestimated (Precipitates < 1 nm cannot be counted);
  Precipitate non-uniform distribution leads to an error.

- **SANS**: (1) Pre-existing small type III precipitates results in a little overestimation;
  (2) Not exactly following lognormal distribution leads to underestimation.

- **Both Methods:** Do not properly consider the precipitates on grain boundaries.

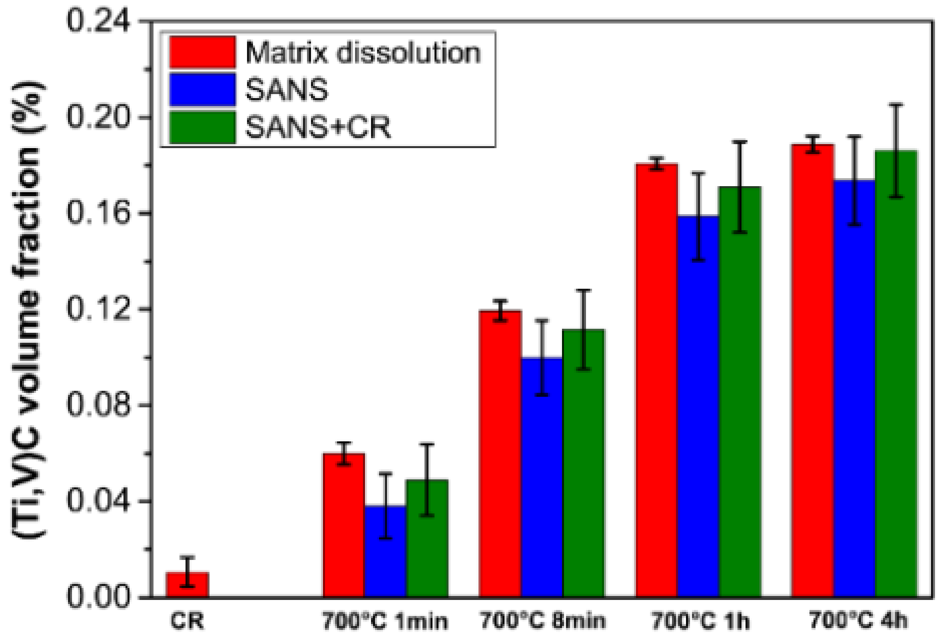
2.6 Precipitate composition comparison

- **TEM-EDS**: Ti/(Ti+V) ratio of individual precipitates;
  ICP-OES: Overall average Ti/(Ti+V) ratio.

Ti/(Ti+V) atomic ratio in type III (Ti,V)C precipitates:

- **TEM-EDS**: 0.6 for 1 nm and decreases to 0.3 for 4 nm precipitates;
- **ICP-OES**: 0.25-0.27.
- The difference is caused by big (Ti,V)C precipitates on grain boundaries.

2.6 Precipitate volume fraction comparison



- Both are underestimated.
- Results of matrix dissolution are comparable to SANS. Difference is that (Ti,V)C volume fraction in the CR sample could not be measured by SANS.
- Matrix dissolution: separate volume fractions of precipitates with different size ranges.

2.7 Precipitate evolution

- **Nucleation**

  Driving force for nucleation higher for TiC than VC. Interface energy is lower for VC.

  TiC-rich core suggests that for nucleation driving force is dominant.

- **Growth**

  Precipitate growth is diffusion controlled. $D_{gb} > D_{dis} >> D_{lattice}$

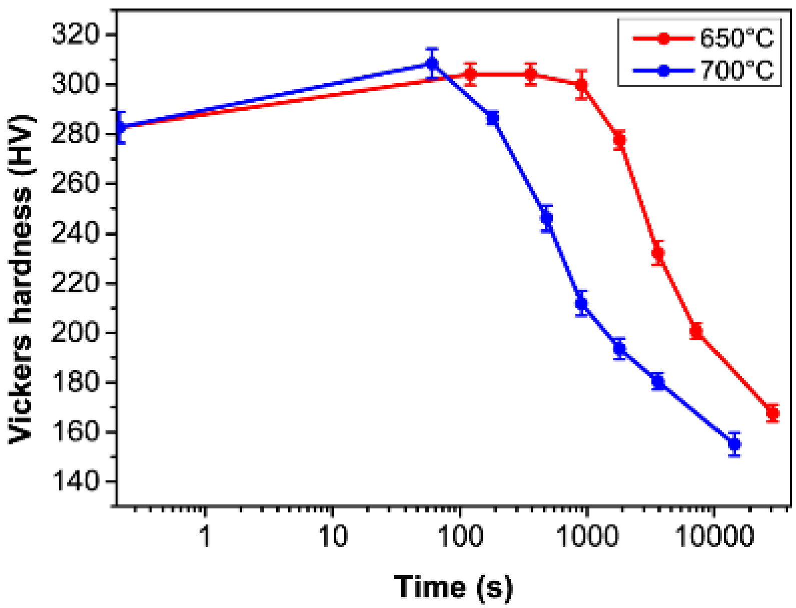
  Precipitates on grain boundaries are bigger.

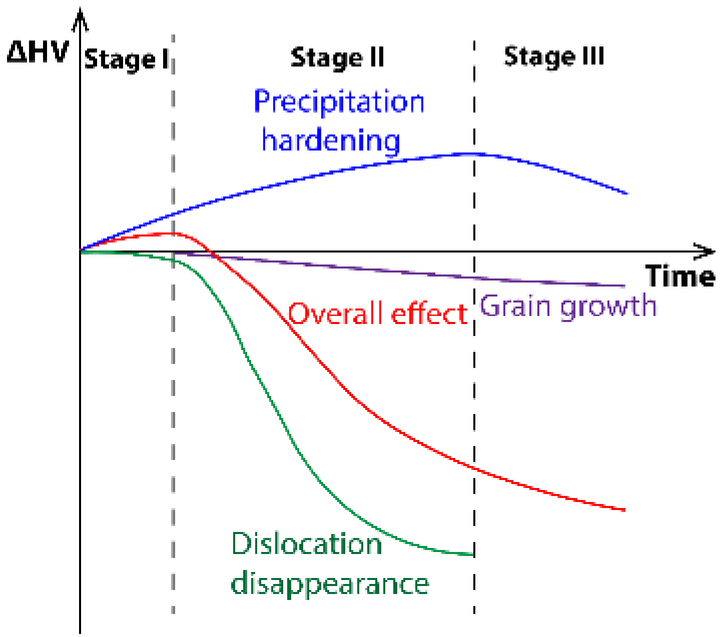
- **Precipitate Lattice parameter**

  Precipitate composition depends on overall steel composition and precipitate size.

  Variations in composition lead to differences in lattice parameter.

2.8 Hardness





Hardness change is a combined result of precipitation, recrystallization and grain growth.

Stage I: Recovery stage (CR sample to 650 °C 6 min and 700 °C 1 min).

Hardness increase dominantly caused by precipitation hardening.

Stage II: Recrystallization stage (650 °C: 6 min to 8 h, 700 °C:1 min to 30 min).

Hardness decrease dominantly caused by recrystallization (dislocation disappearance).

Stage III: Grain growth stage (700 °C: 30 min to 4 h).

Hardness decrease is caused by precipitate coarsening and grain growth.

3. Conclusions

(1) Unique combination of precipitate quantification methods has been used to analyze Ti-V HSLA steel.

(2) Recrystallization nearly completes after annealing at 650 °C for 8 h, while it takes 30 min at 700 °C.

(3) Three types of precipitates were identified in all the samples: large Ti(C,N), medium (Ti,V)(C,N) and small spherical (Ti,V)C. The first two types are stable while the third type evolve during annealing.

(4) The Ti/(Ti+V) ratio in (Ti,V)C precipitates decreases with increasing precipitate size. The annealing temperature or time has no observable effect on this ratio.

(5) Precipitate size increases with the annealing temperature and time. The mean size obtained by TEM is bigger than the one of SANS measurement.

(6) Precipitate volume fraction in steels were obtained by both SANS and matrix dissolution. The (Ti,V)C volume fractions obtained by both methods are in close agreement. Matrix dissolution method allows separate determination of the volume fraction of the bigger pre-existing (Ti(C,N) and (Ti,V)(C,N)) precipitates and the smaller (Ti,V)C precipitates.

(7) The hardness first increases and then decreases when annealing at both 650 °C and 700 °C. The hardness increase is dominated by precipitation while the decrease is mainly caused by recrystallization.