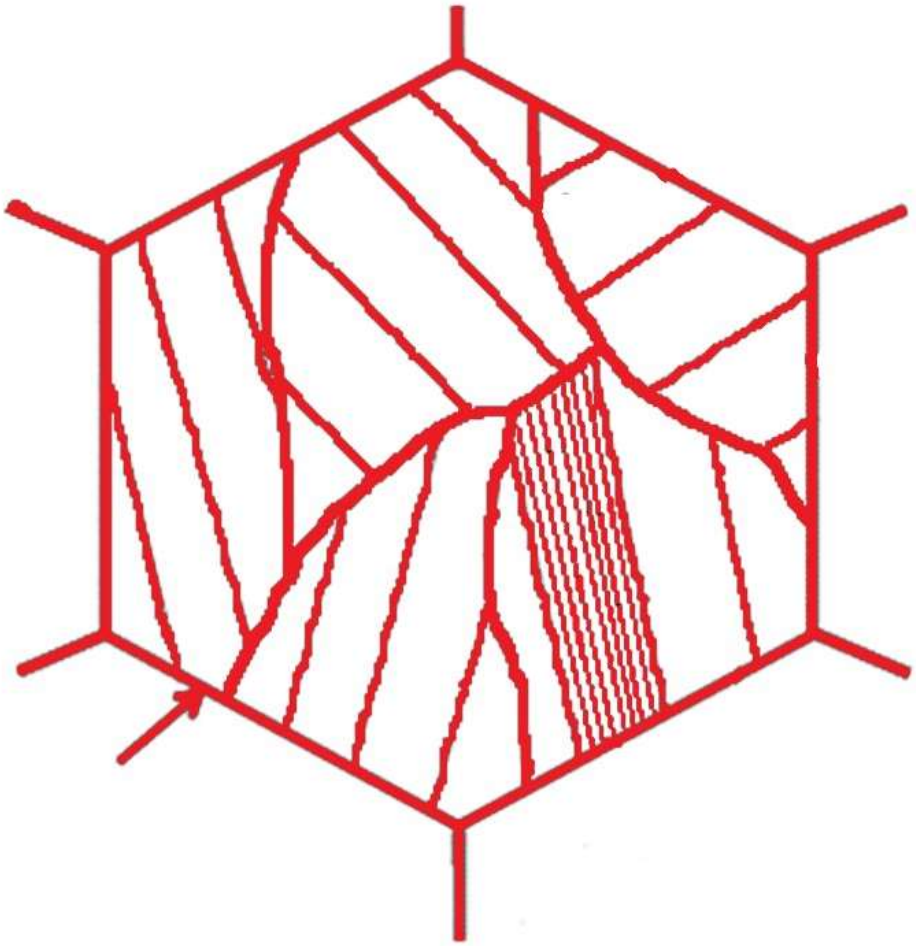
Neutron-diffraction Studies of Structural-Phase Transitions in Alloys

T.N. Vershinina, S.V. Sumnikov, I.A. Bobrikov, R.N. Vasin, N.Yu. Samoylova,

A.M. Balagurov

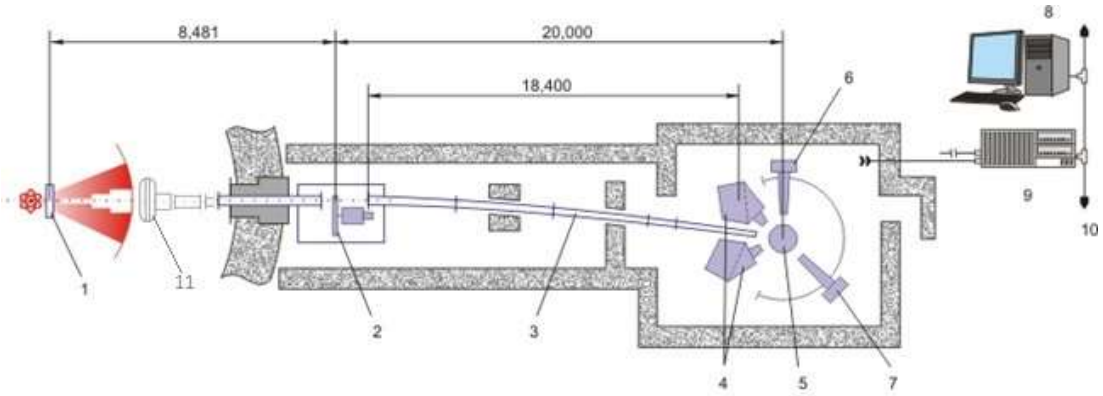


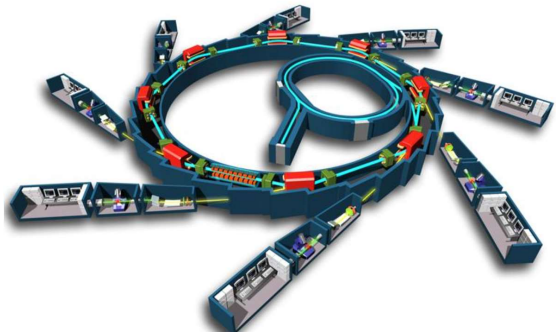
Elements of the microstructure that should be controlled during development of alloys with desired characteristics:

- **phase composition of main phases**
- **phase composition of secondary phases**
- **grain boundary ensemble**
- **dislocation density**
- **microstructure elements dimensions**
- **etc.**

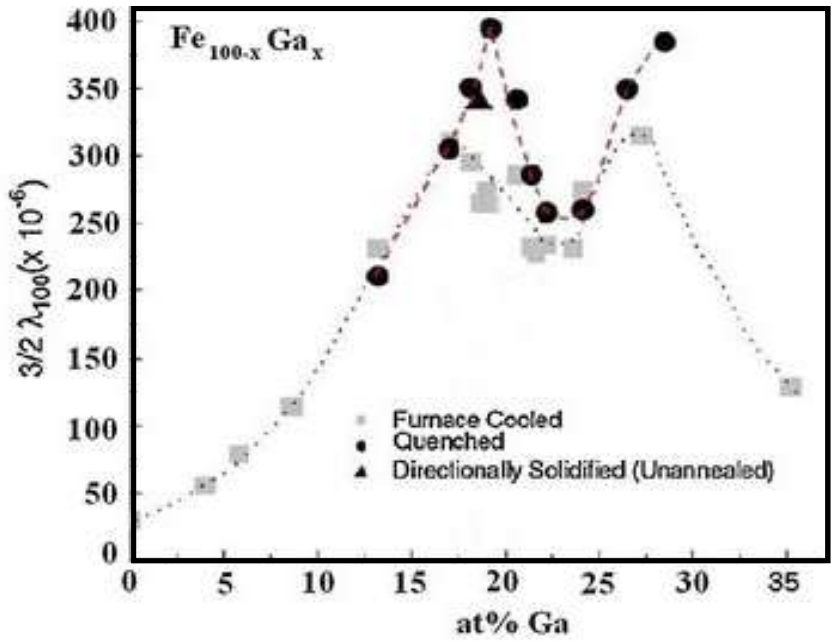
The main diffraction methods for structure and phase composition studying

- Diffraction of electrons
- X-ray diffraction
- Diffraction of synchrotron radiation
- Neutron diffraction





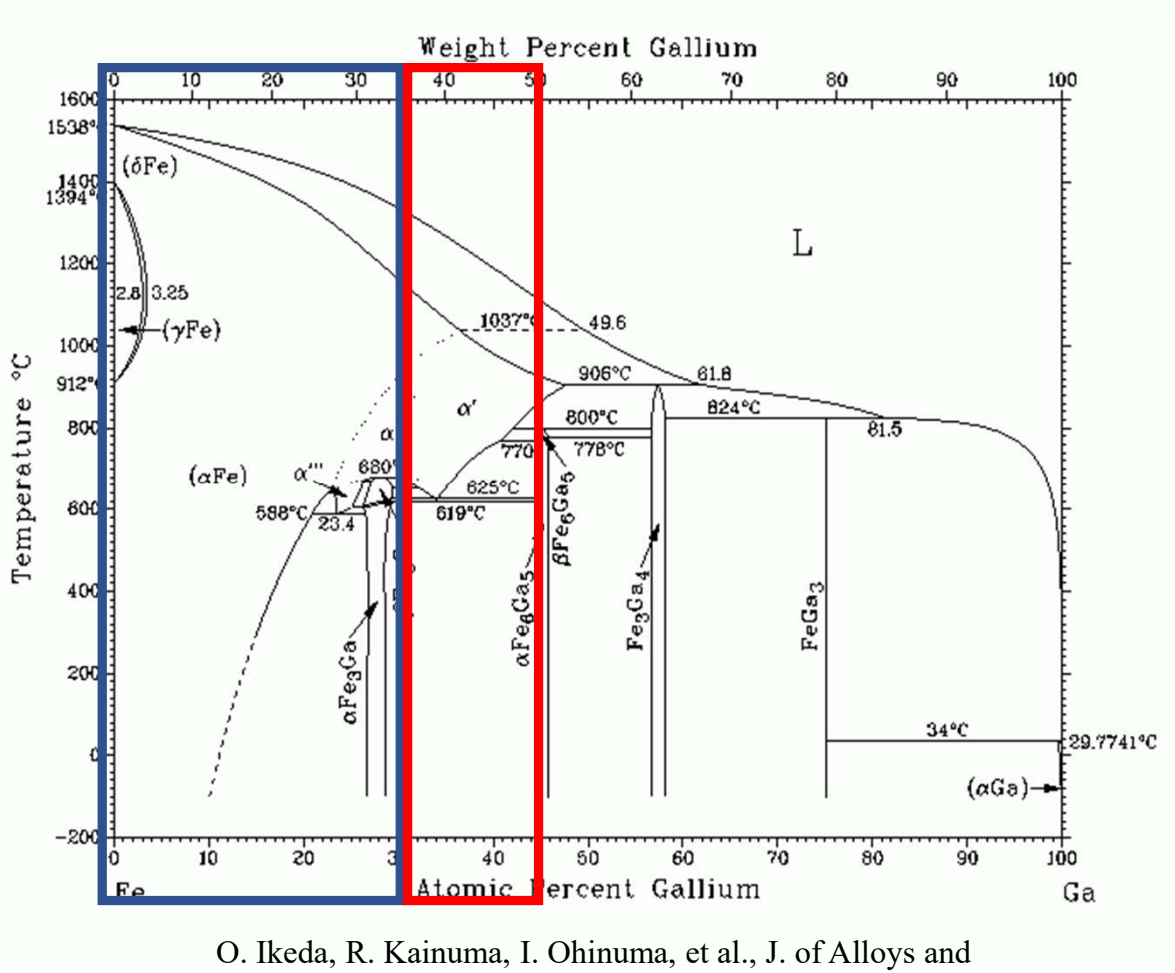
Giant magnetostriction in Fe-Ga alloys



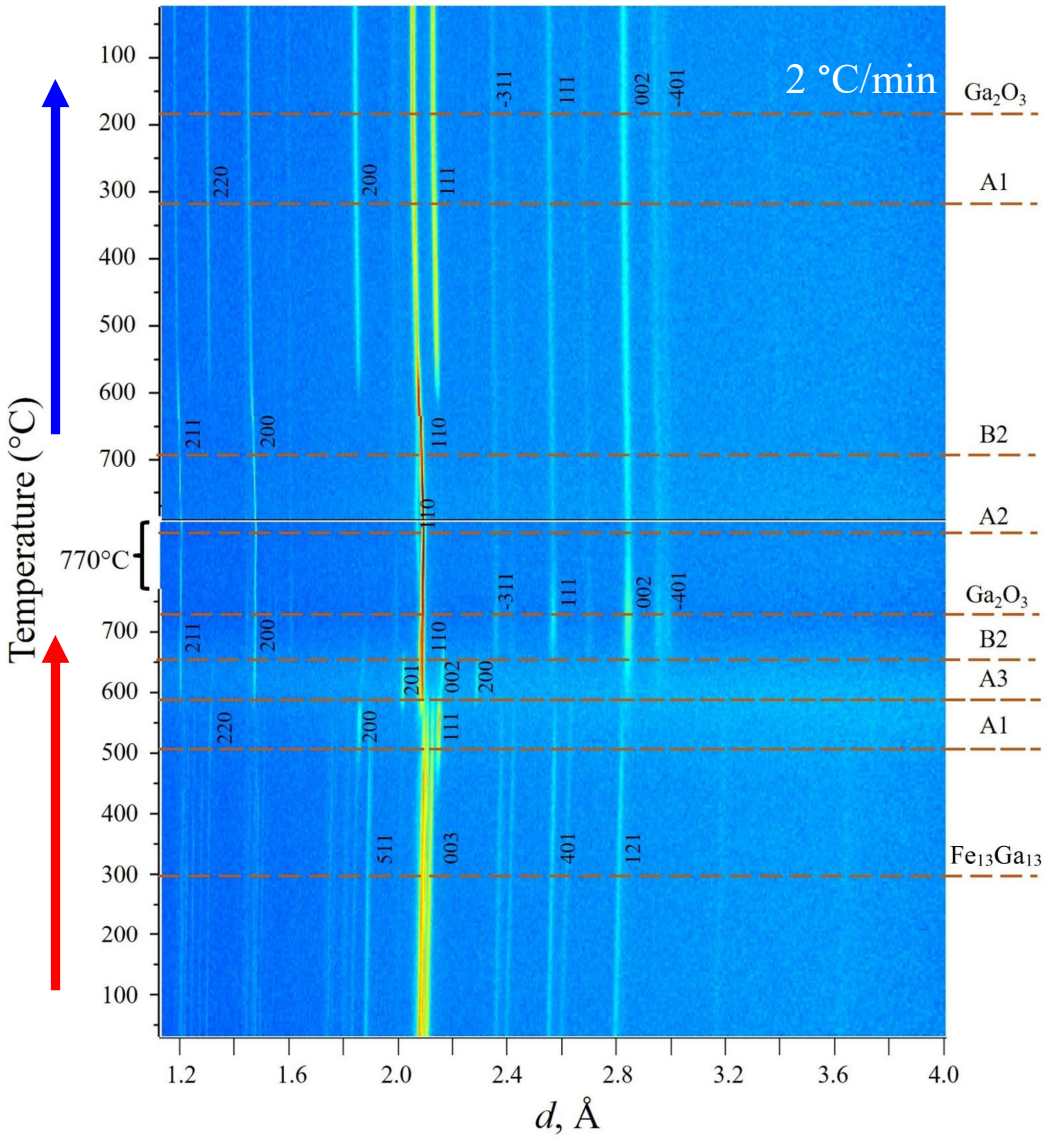
T.A. Lograsso, E.M. Summers Materials Science and Engineering (2006) A 416 240

	$\lambda \sim \Delta l/l$:	
Fe	**$\sim 20\times10^{-6}$**	
Fe-Al	**$\sim 60\times10^{-6}$**	
Fe-19Ga	**$\sim 400\times10^{-6}$**	**! ?**

A.E. Clark et al. IEEE Transactions on Magnetics (2000) , Vol. 36, Issue 5

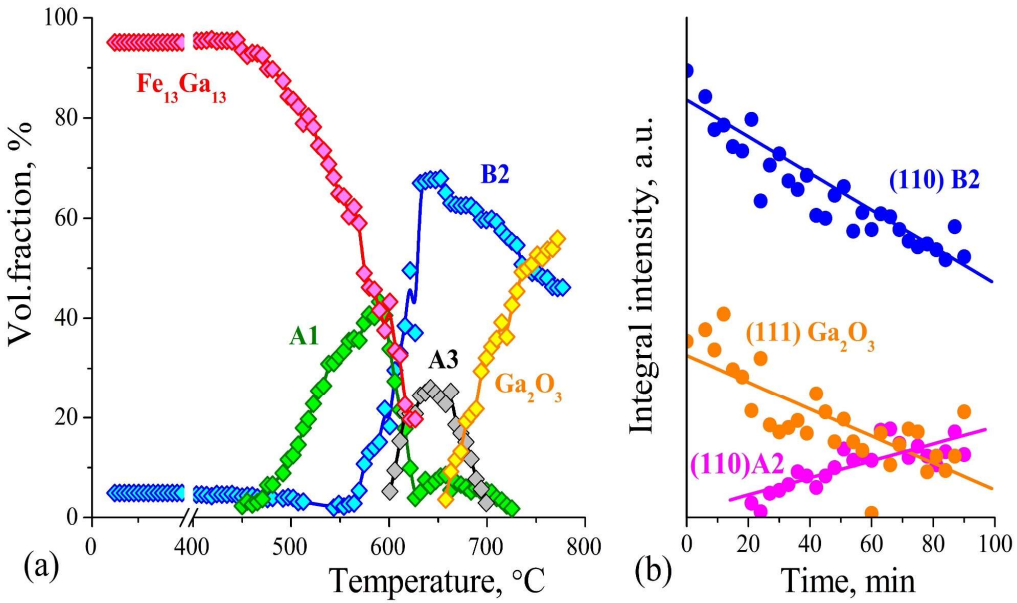


O. Ikeda, R. Kainuma, I. Ohinuma, et al., J. of Alloys and Compounds 347 (2002) 198.



2D visualization of the evolution of neutron diffraction patterns

Phase transformations in the Fe-45Ga alloy during heating to 770 °C and subsequent cooling (XRD)

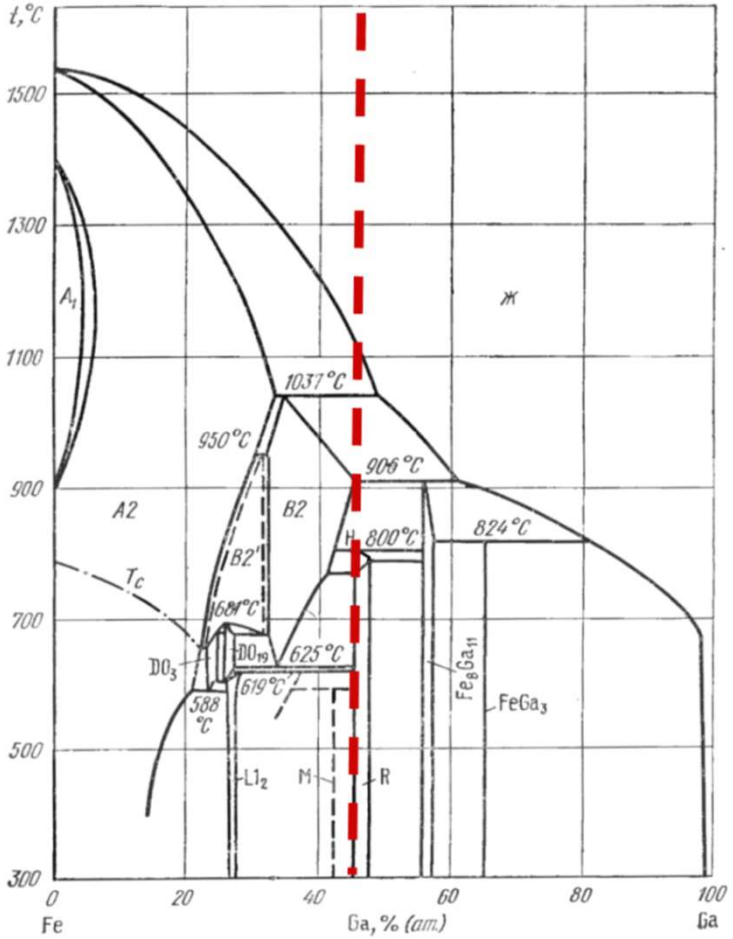


Heating up to 770°C (a) and subsequent isothermal exposure for 90 minutes (b) (XRF).

Vershinina T.N. et.al. Intermetallics, 2021, 131 (1–5), 107110

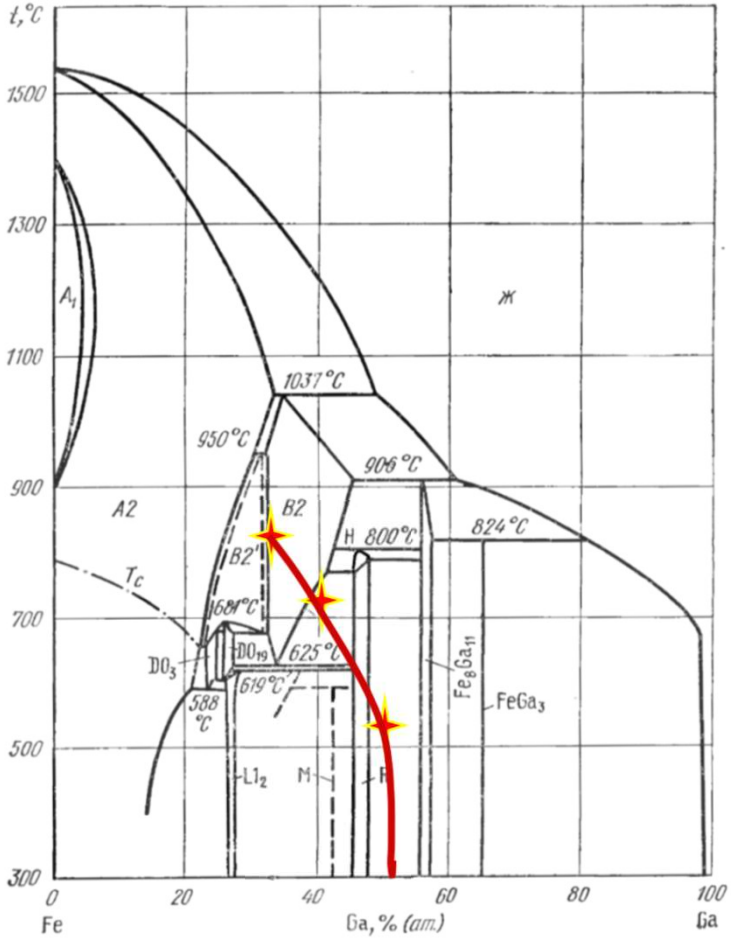
Expectation

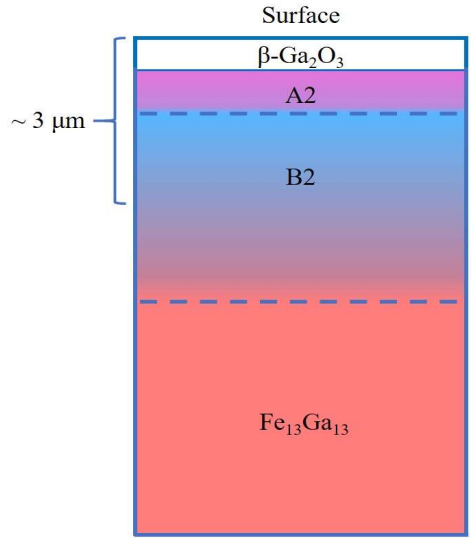
$$Fe_{13}Ga_{13} + B2 \xrightarrow{x°C} \alpha\text{-}Fe_6Ga_5 + B2 \xrightarrow{\sim 770°C} Fe_{13}Ga_{13}$$



Reality

$$Fe_{13}Ga_{13} + B2 \xrightarrow{450°C} B2 + L1_2 \xrightarrow{600°C} B2 + D0_{19}$$
$$\xrightarrow{650°C} B2{+}A2 + Ga_2O_3$$

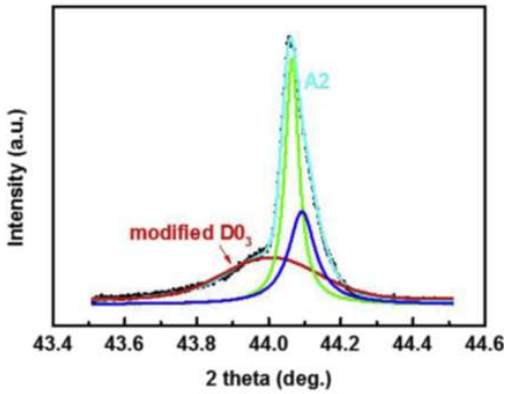




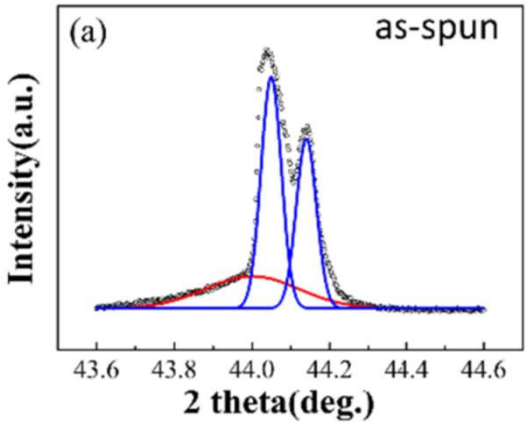
Schematic representation of the surface layer of the Fe–45.0 Ga alloy after heating to 770°C

Fe-27Ga
m-$D0_3$

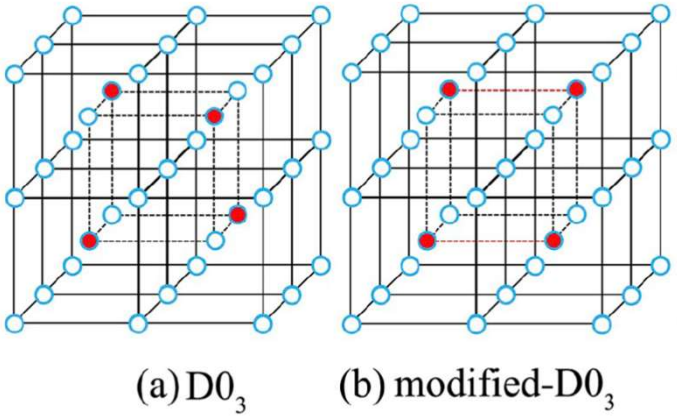
Synchrotron XRD diffraction



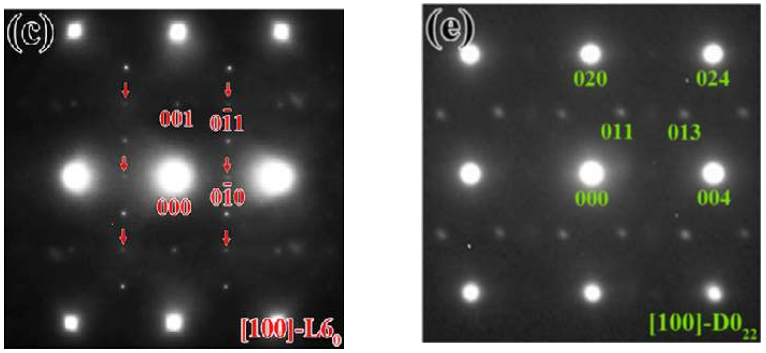
Y. He, et al., Acta Mater. 109 (2016) 177-186.



Y. Han, et al., Scr. Mater. 150 (2018) 101–105.

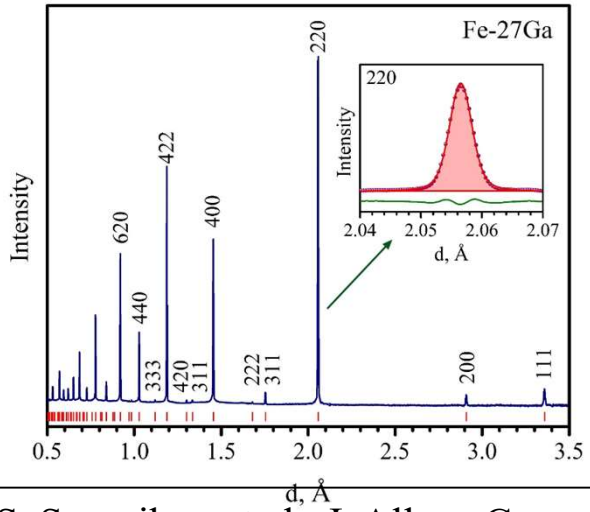


Electron diffraction

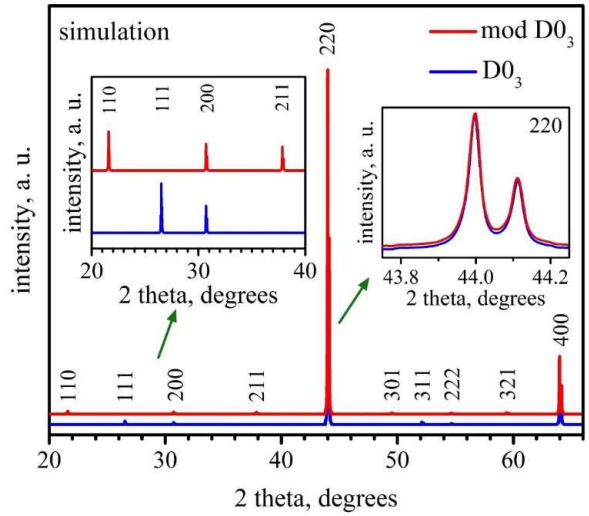


J. Gou et al, Scr. Mater. 185 (2020) 129–133.

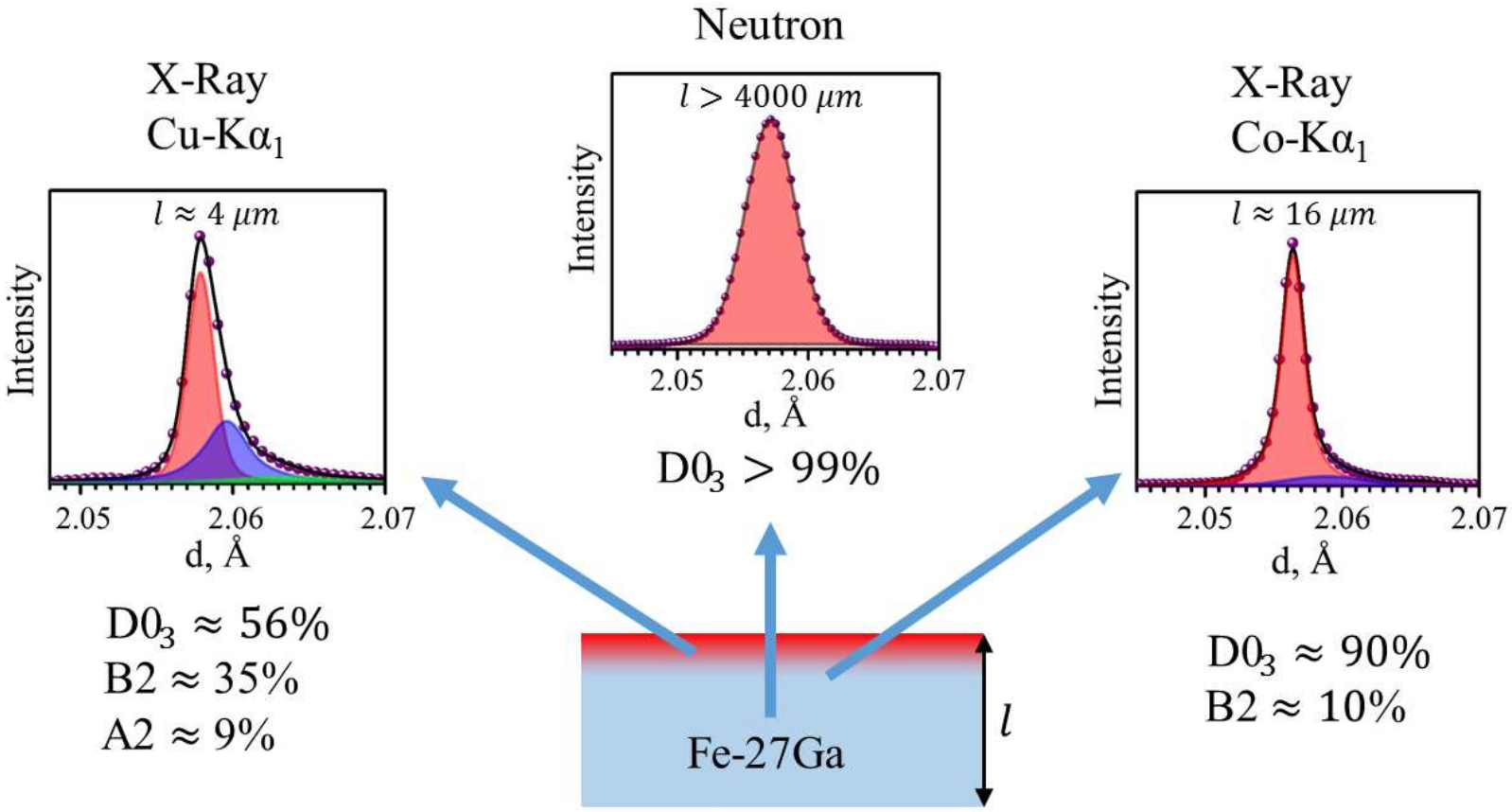
Neutron diffraction



S. Sumnikov et al, J. Alloys Compd. 928 (2022) 167116

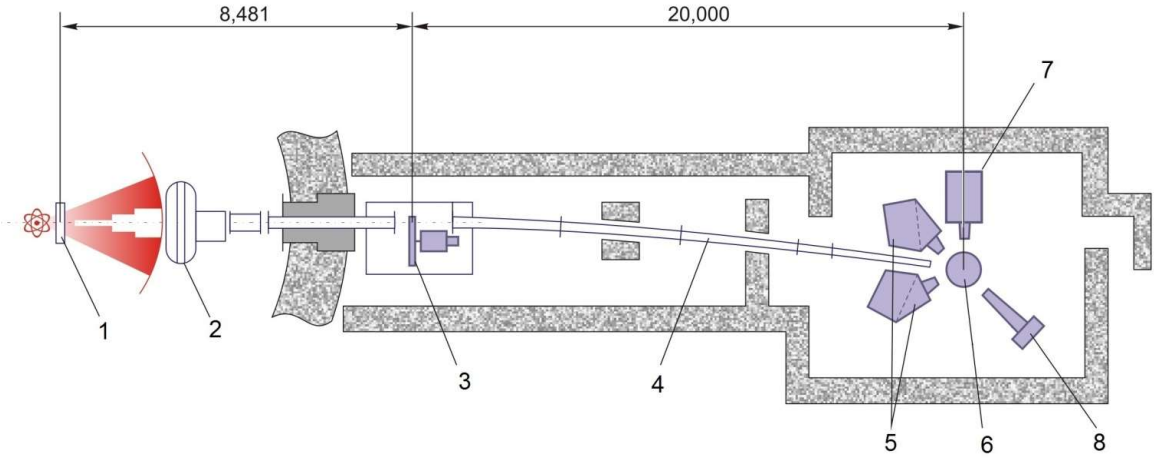


m-$D0_3$ существует или нет?



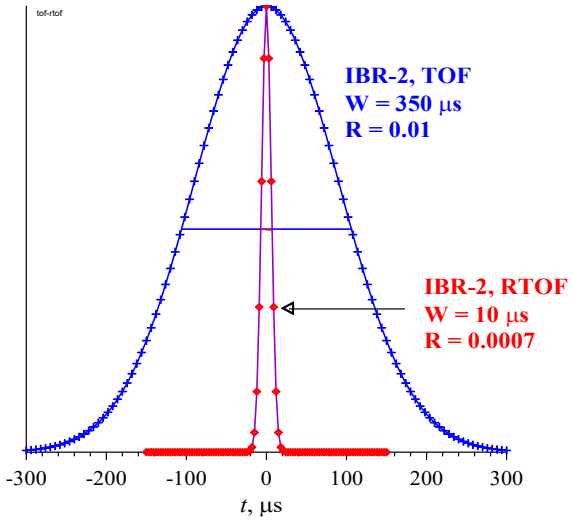
S. Sumnikov et al, J. Alloys Compd. 928 (2022) 167116

HRFD – High Resolution Fourier Diffractometer at IBR-2



Neutron diffraction:

- large penetration depth;
- the ability to distinguish elements that are closely located in the periodic table;
- large cross section of the neutron beam;
- ability of neutrons to feel the magnetic structure of the material.



High-resolution mode:

$\Delta t_0 \approx 10$ μs, $\Delta d/d \approx$ **0.0015** for $d = 2$ Å

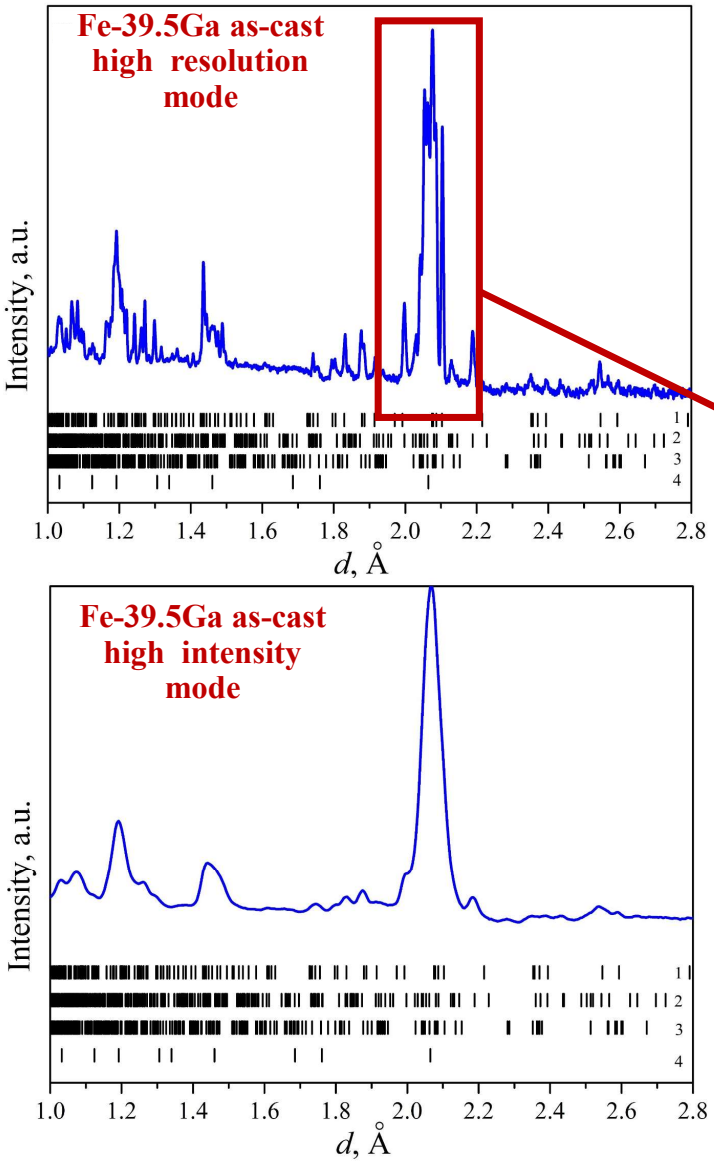
$\Phi_0 \approx 4 \cdot 10^6$ n/cm^2/s, $t_s \sim$ **1 h**

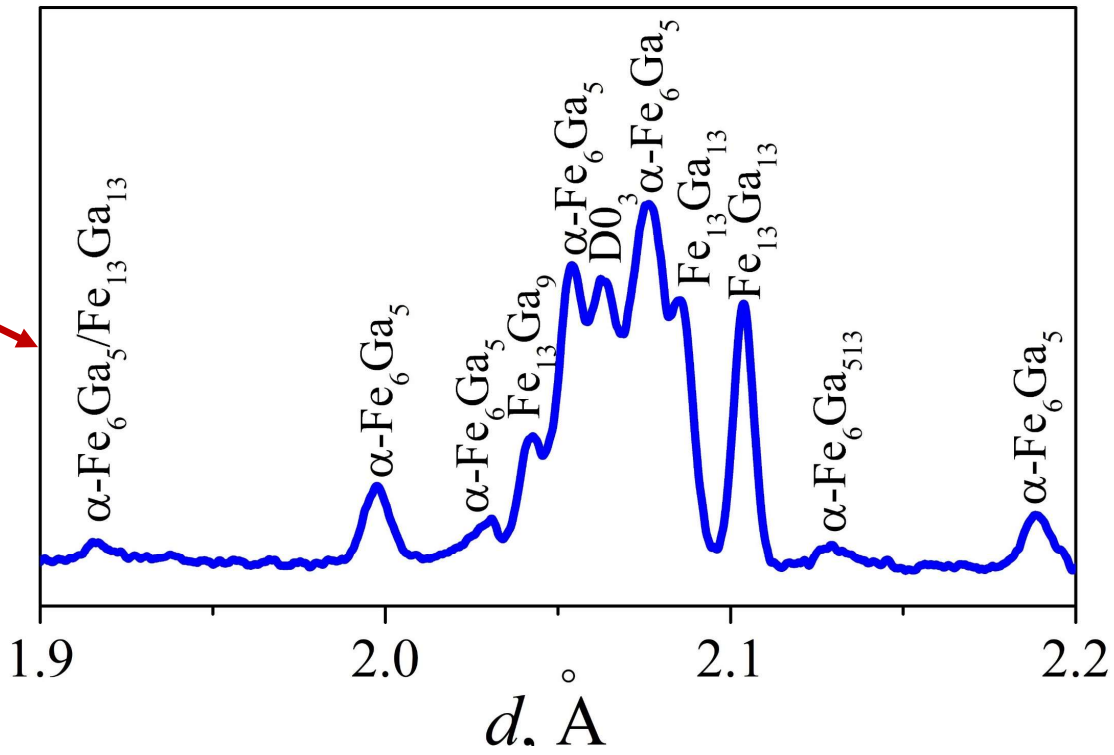
High-intensity mode:

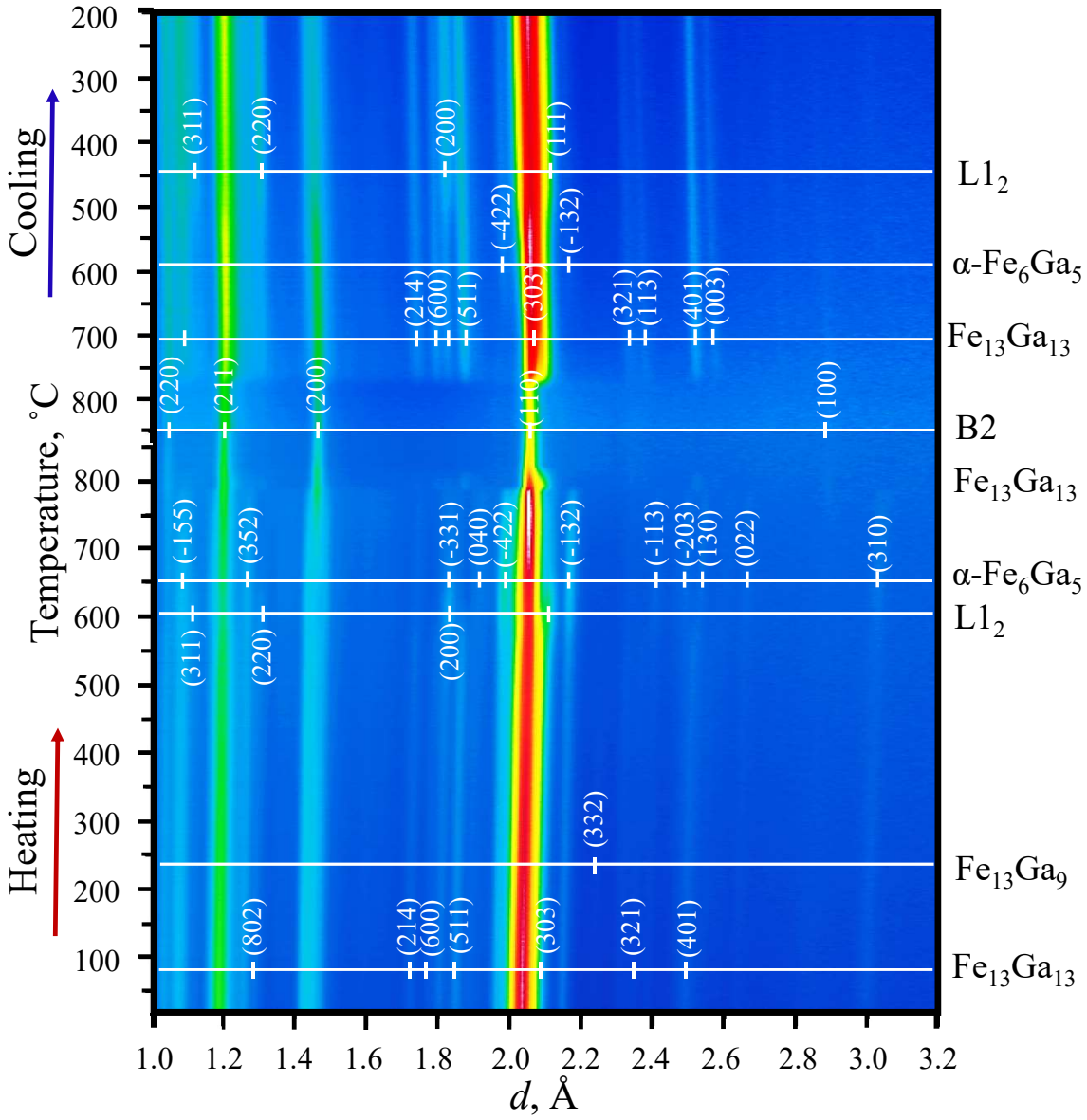
$\Delta t_0 \approx 350$ μs, $\Delta d/d \approx$ **0.015** for $d = 2$ Å

$\Phi_0 \approx 2 \cdot 10^7$ n/cm^2/s, $t_s \sim$ **1 min**

High-resolution and high-intensity modes







2D visualization

2D visualization of the evolution of neutron diffraction patterns of the Fe-39.5Ga sample in the cast state. Diffraction patterns was measured during slow heating to 850 ºC. The temperature (and time) axis goes from bottom to top. Heating and cooling were carried out at a rate close to 2 °C/min. The dashes indicate the peak positions of the phases indicated on the right.

BCC phases: A2 (sp.gr. $Im\bar{3}m$),
B2(sp.gr. $Pm\bar{3}m$),
D0$_3$ (sp.gr. $Fm\bar{3}m$)

L1$_2$ (sp.gr. $Pm\bar{3}m$)

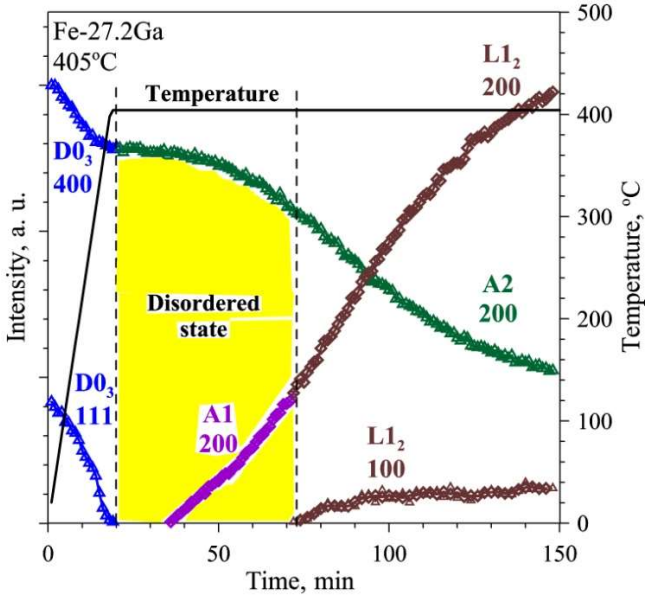
α-Fe$_6$Ga$_5$ (sp.gr. $C2/m$)

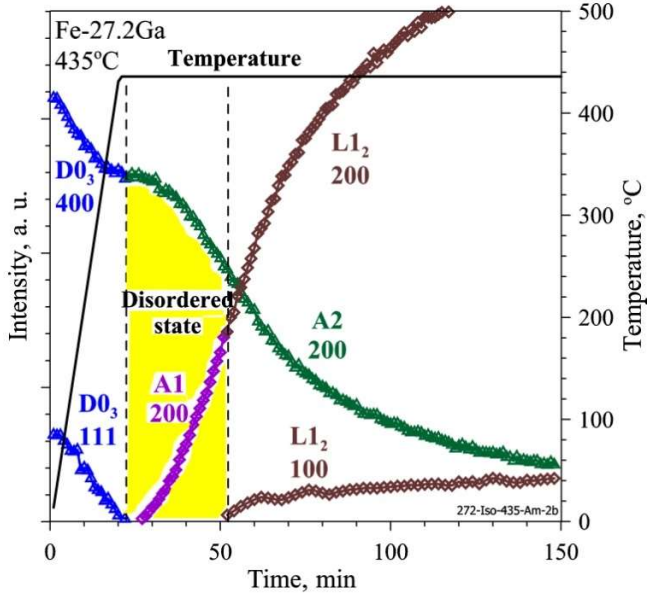
Fe$_{13}$Ga$_9$ (sp.gr. $C2/m$)

Fe$_{13}$Ga$_{13}$ (sp.gr. R-3m)

Mechanisms of phase transformations in Fe-Ga alloys

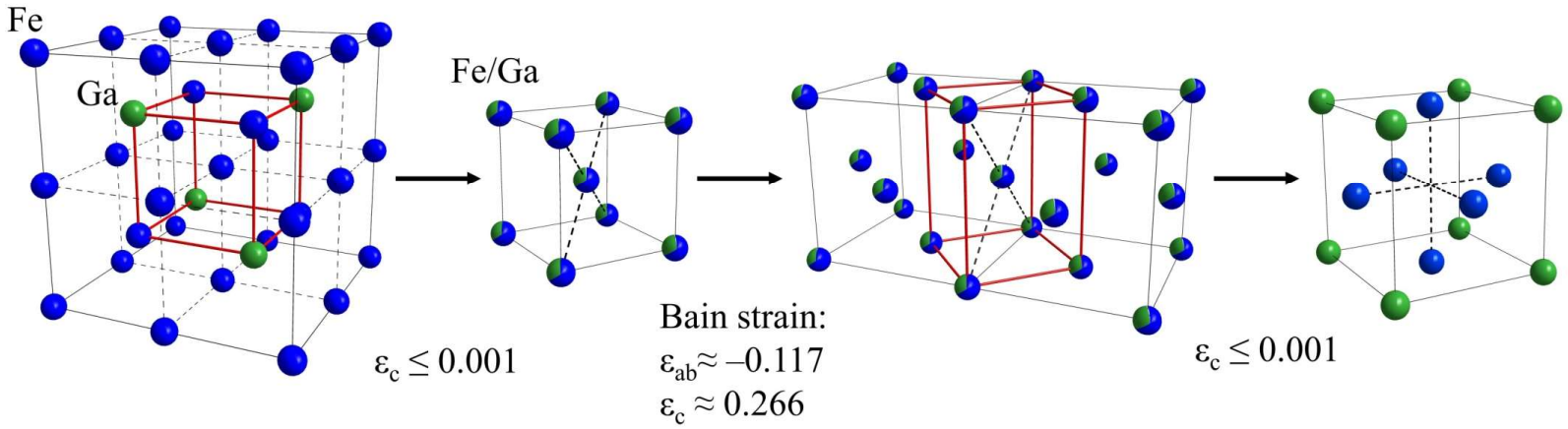
Complex phase transition combining the diffusional and displacive stages in Fe-27Ga





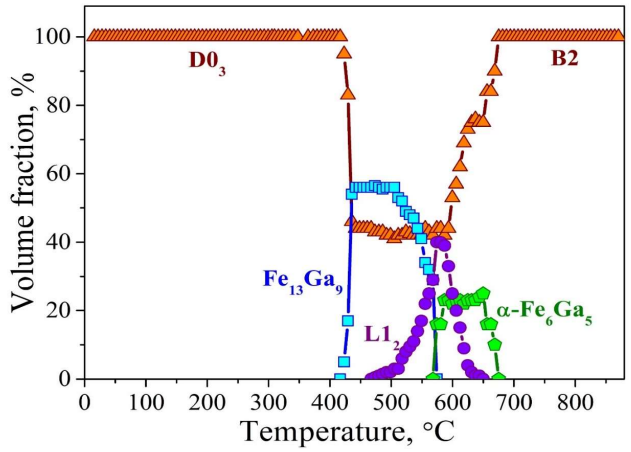
Balagurov A.M. et.al. Acta Cryst. (2019). B75, 1024-1033

$D0_3$ **diffusional →** A2 **displacive →** A1 **diffusional →** $L1_2$

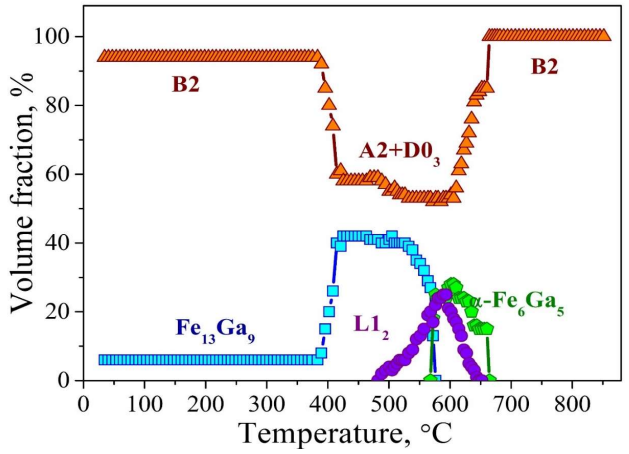


Evolution of the phase composition of Fe-(31-39)Ga during heating up to 850 °C

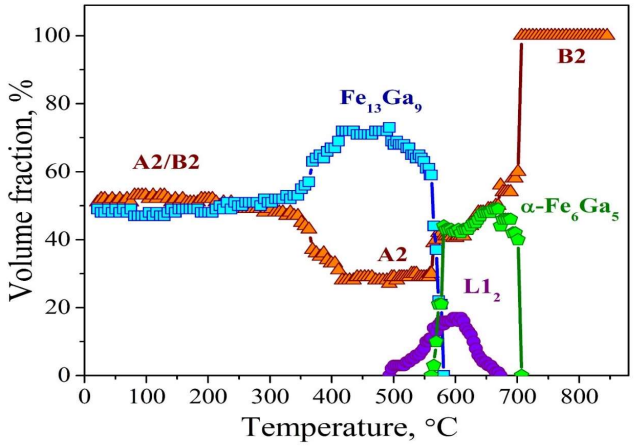
Fe-31.1Ga



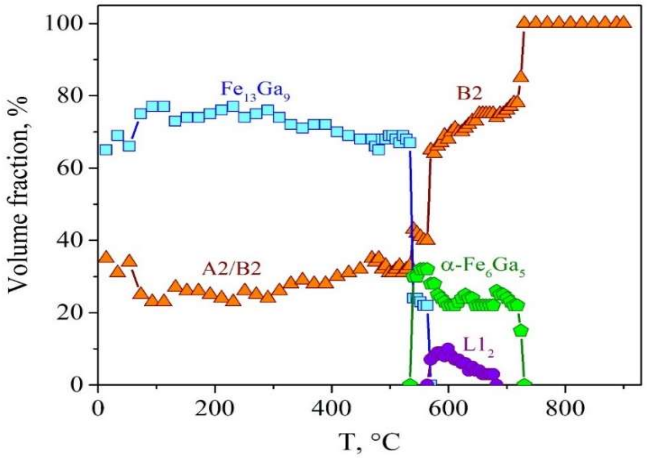
Fe-34.4Ga



Fe-32.9Ga



Fe-38.4Ga

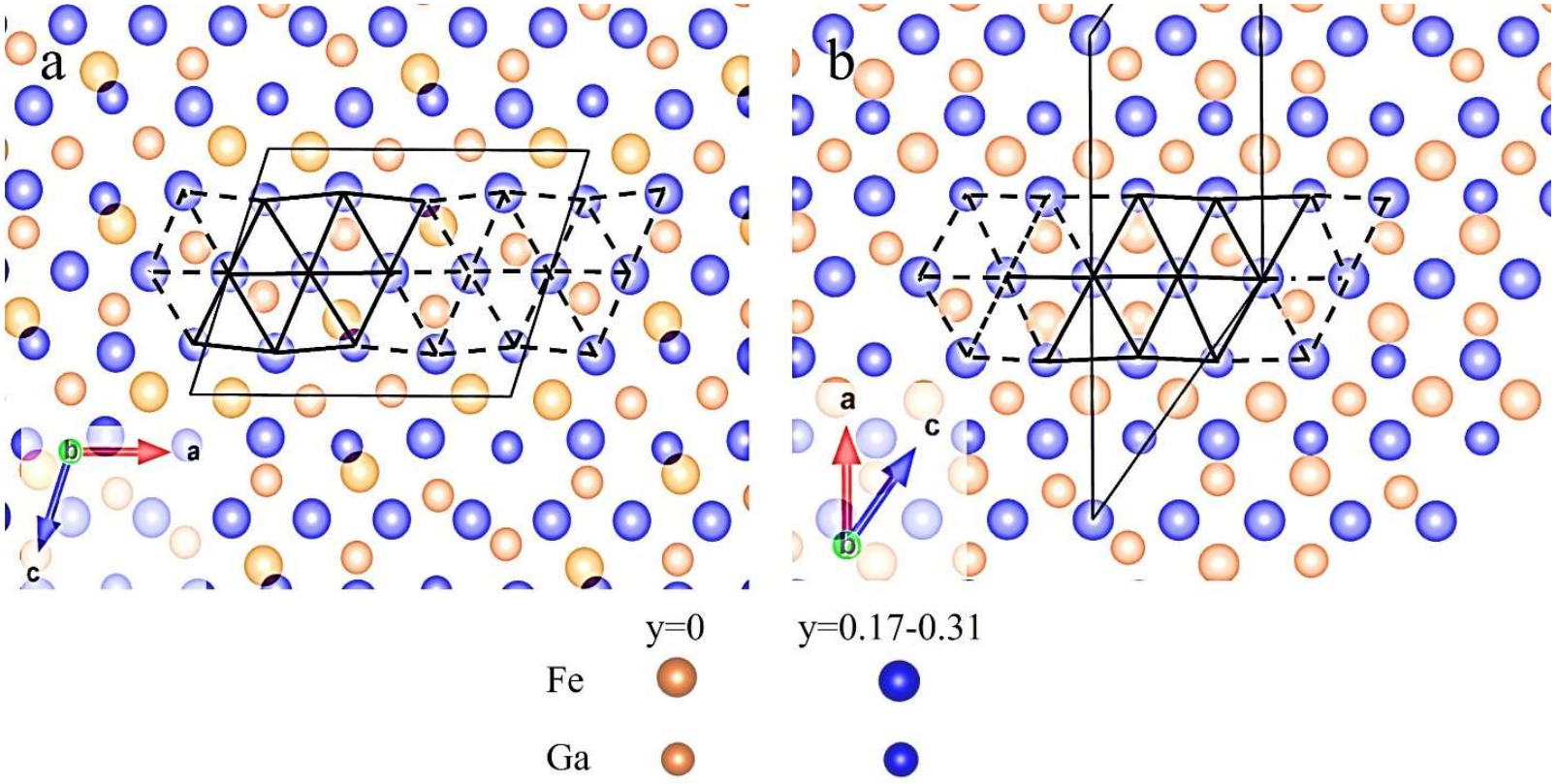


Vershinina T.N. et.al., Journal of Alloys and Compounds, 889 (2022) 161782
Vershinina T.N et.al., Journal of Alloys and Compounds 934 (2023) 167967

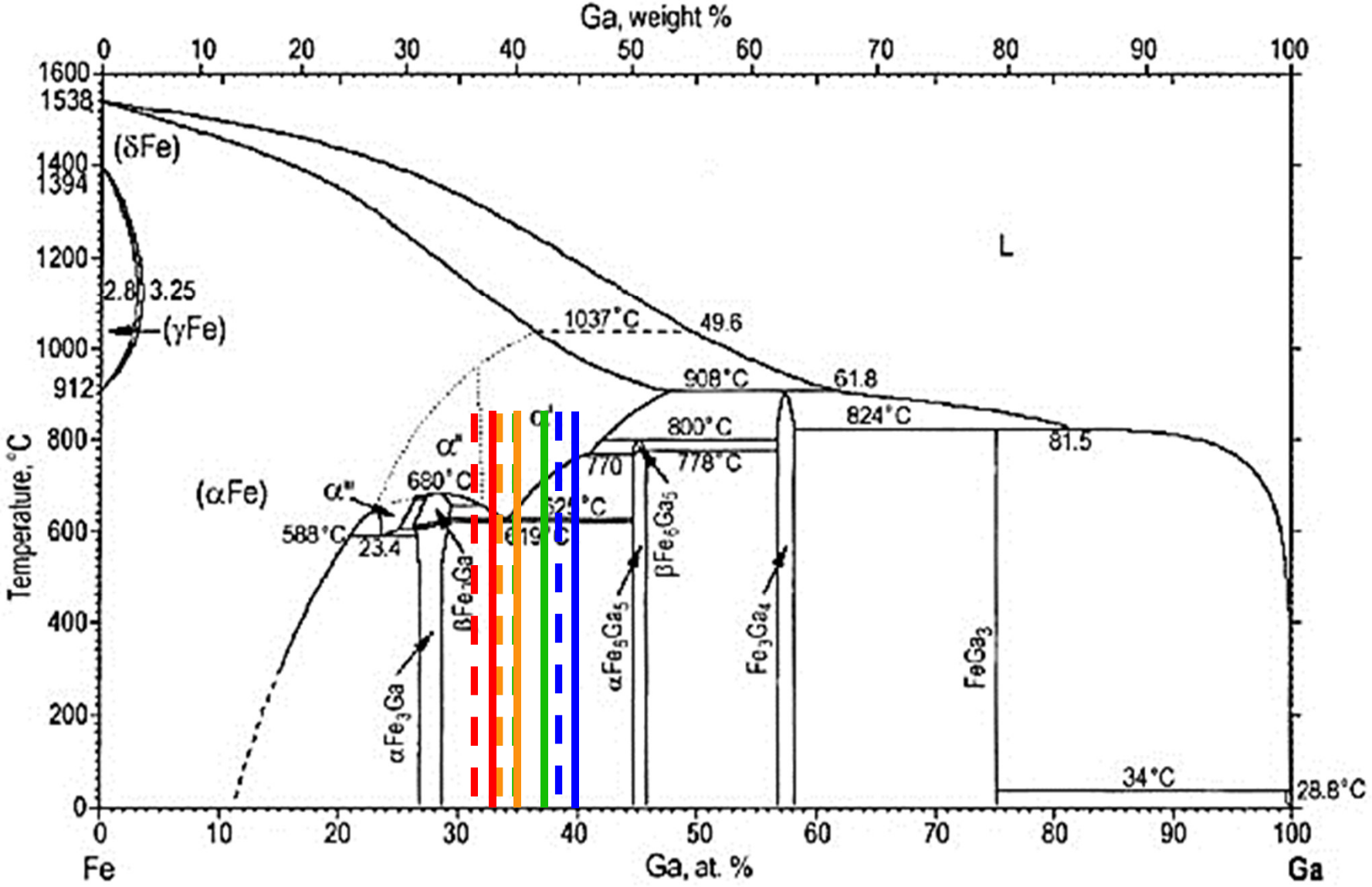
$Fe_{13}Ga_9 \rightarrow L1_2 + \alpha\text{-}Fe_6Ga_5$

$\alpha\text{-}Fe_6Ga_5$ (sp.gr. *C*2/*m*)

$Fe_{13}Ga_9$ (sp.gr. *C*2/*m*)



Фазовый состав сплавов Fe-(31-39)Ga в as-cast состояниях



Summary

- **Neutron diffraction provides information from a large volume of samples. The influence of surface layers on the results obtained is excluded.**

- **The combination of measurements at room temperature in high resolution mode and *in situ* measurements in high intensity mode reveals both the order of phase transformations and the mechanisms of these phase transformations.**

Thank you for attention!