

Aluminium base amorphous and nanocrystalline alloys with Fe impurity

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Al based materials → more than 90 % Al

Rapid quenching methods → **Al** → new microstructures and property enhancements

Mechanical properties → partial crystallization
→ nanostructured materials

Base → Al – TM – RE

↓ ↓

Transition metals Rare earth

Ways

1) Undercooling and crystallization

Al intermetallic phases, nanocrystalline Al in amorphous rest

2) Mechanical alloying

Provides insight in nanocrystalline structure and amorphisation

Type of samples

1) Fe – Sm, Fe – Y, Fe – Ni → first works

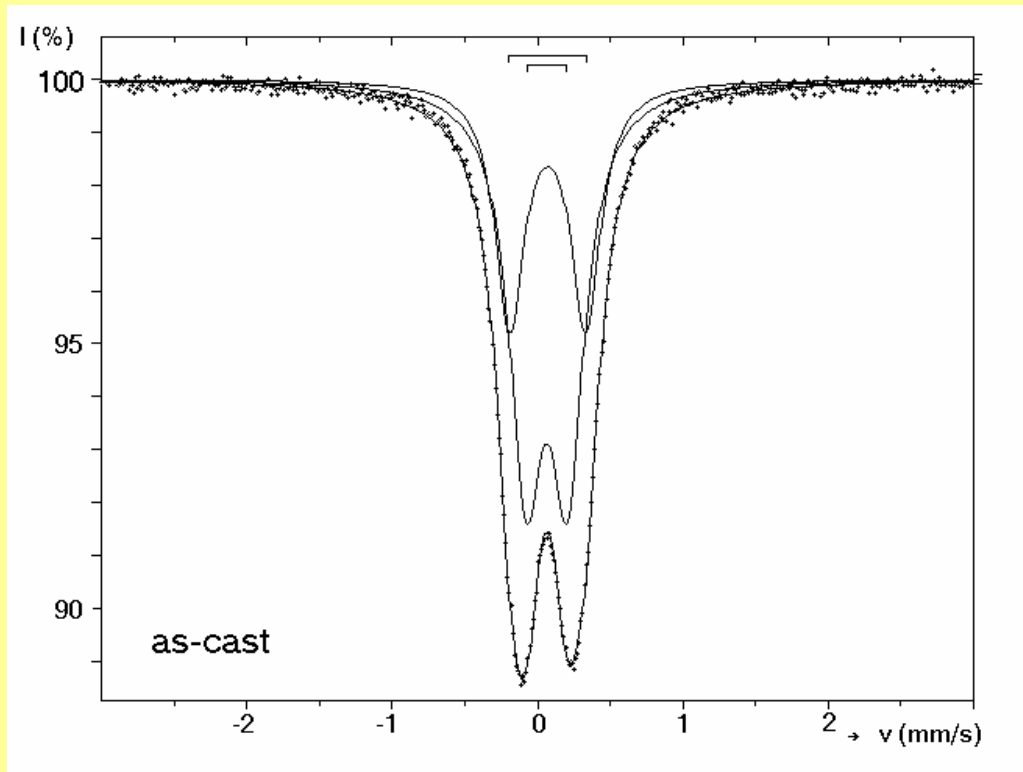
2) Fe – Nb → phase diagram Al – Nb is similar to Al – rare earth

Our samples $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$ and $\text{Al}_{94}\text{Fe}_2\text{V}_4$

Planar flow casting – Institute of Physics,
Slovak Academy of Science

As-cast samples

Sample of $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$



Amorphous Al-Fe

$\delta=0.18$ mm/s

$\Delta=0.52$ mm/s

***Fadeeva et. al: Mat. Sc. Eng. A206
(1996) 90-94***

Two cluster model

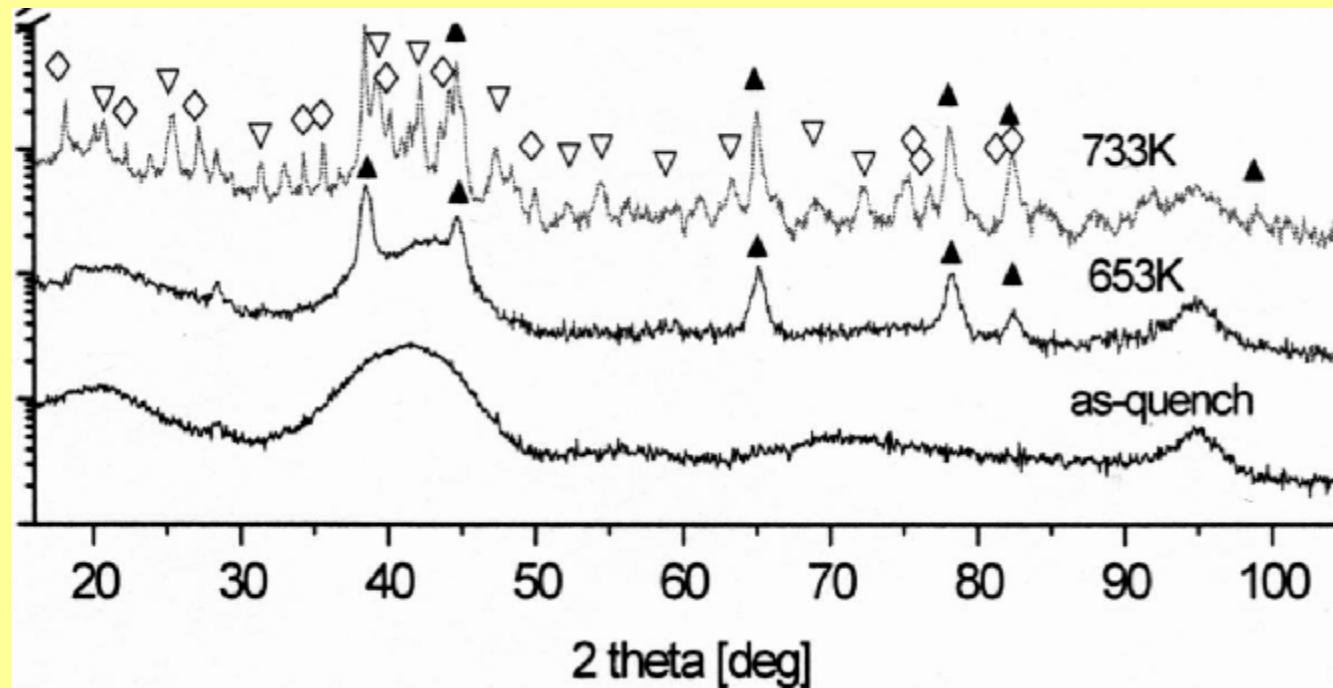
Al clusters, Al-Fe-Nb clusters

***Švec et.al Mat. Sc. Eng. A375-377
(2004) 946-950***

Our results: $\delta=0.18$ mm/s, $\Delta=0.52$ mm/s, $A=63\%$

$\delta=0.17$ mm/s, $\Delta=0.29$ mm/s, $A=37\%$

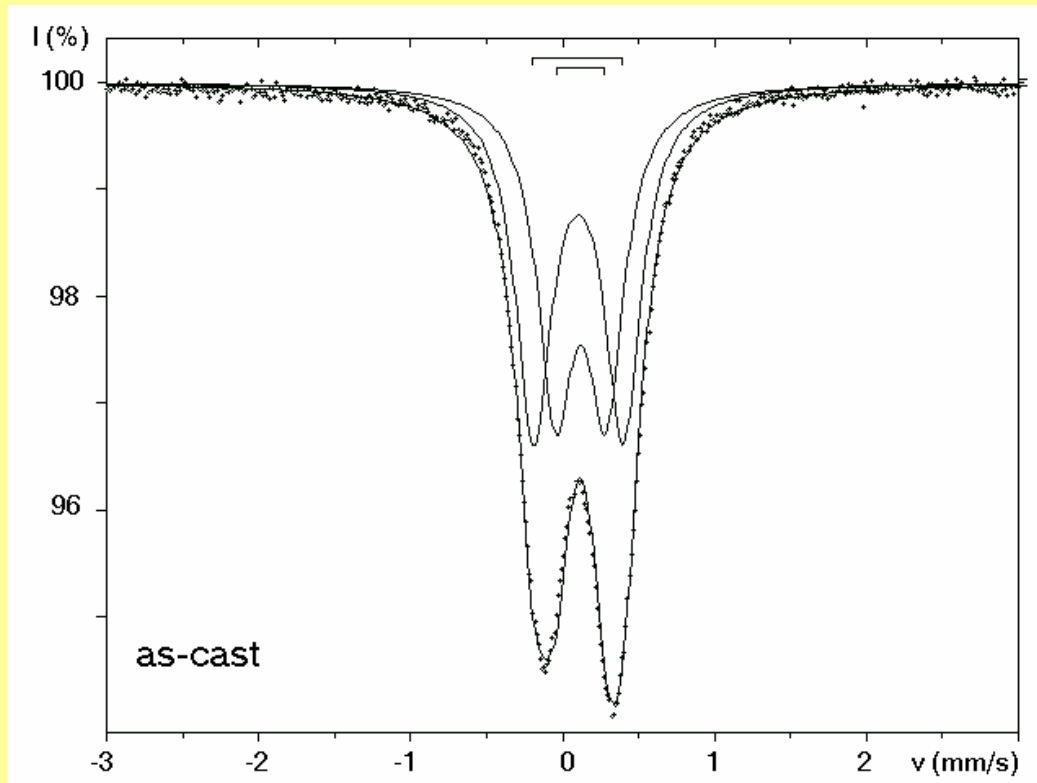
XRD spectrum of $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$



- \triangle ... Al_3Nb
- \blacktriangle ... Al
- \diamond ... Al_3Fe

As-cast samples

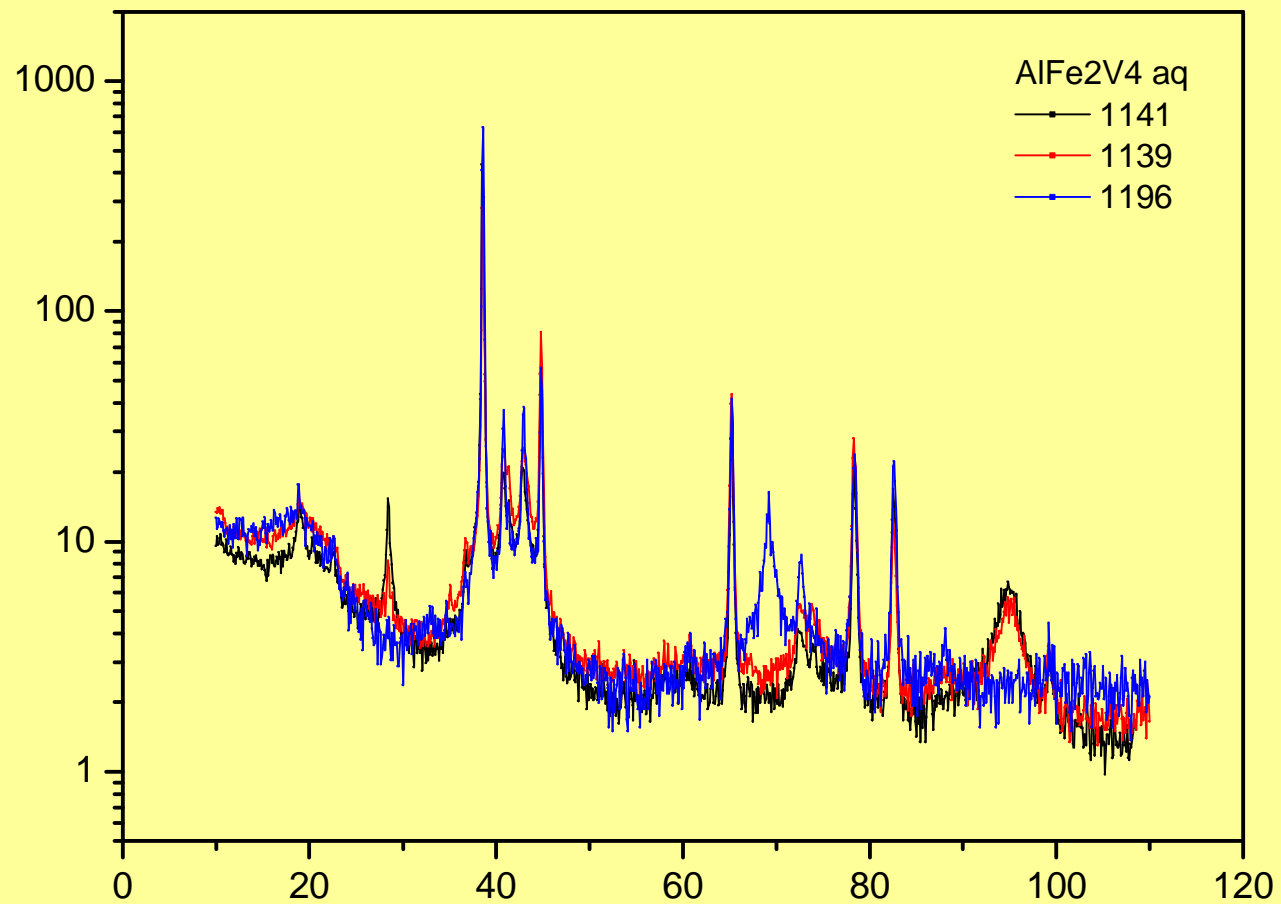
Sample of $\text{Al}_{94}\text{Fe}_2\text{V}_4$



This sample is not fully amorphous

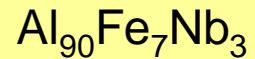
Our results: $\delta=0.21$ mm/s, $\Delta=0.59$ mm/s, $A=53\%$
 $\delta=0.22$ mm/s, $\Delta=0.33$ mm/s, $A=47\%$

XRD spectrum of $\text{Al}_{94}\text{Fe}_2\text{V}_4$

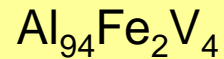


Lamb – Mössbauer factor

Area method was used to determine f-factor



$$f=0.26 \pm 0.02$$



$$f=0.31 \pm 0.02$$

Debye temperature was calculated using classical formula:

$$f = \exp \left\{ -\frac{6E_R}{k_b \Theta_D} \cdot \left[\frac{1}{4} + \left(\frac{T}{\Theta_D} \right)^2 \cdot \int_0^{\frac{\Theta_D}{T}} \frac{x}{e^x - 1} dx \right] \right\}$$

$$\Theta_D=296 \pm 5 \text{ K}$$

$$\Theta_D=318 \pm 5 \text{ K}$$

For the $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$ was Debye temperature derived from measurement of second order Doppler shift as $\Theta_D=325 \pm 5 \text{ K}$

The effective Debye temperature for binary system is:

$$\Theta_{eff} = \Theta_{host} \sqrt{\frac{M_{host}}{M_{imp}}} \sqrt{\frac{\lambda_{imp}}{\lambda_{host}}}$$

λ ...force constant, usually $\frac{\lambda_{imp}}{\lambda_{host}} \approx 1$

For pure aluminium: $\Theta_D=428$ K

For iron impurity in aluminium:

$$\Theta_{eff}=295$$
 K

Close to Debye temperature of as-cast $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$

Crystallization process

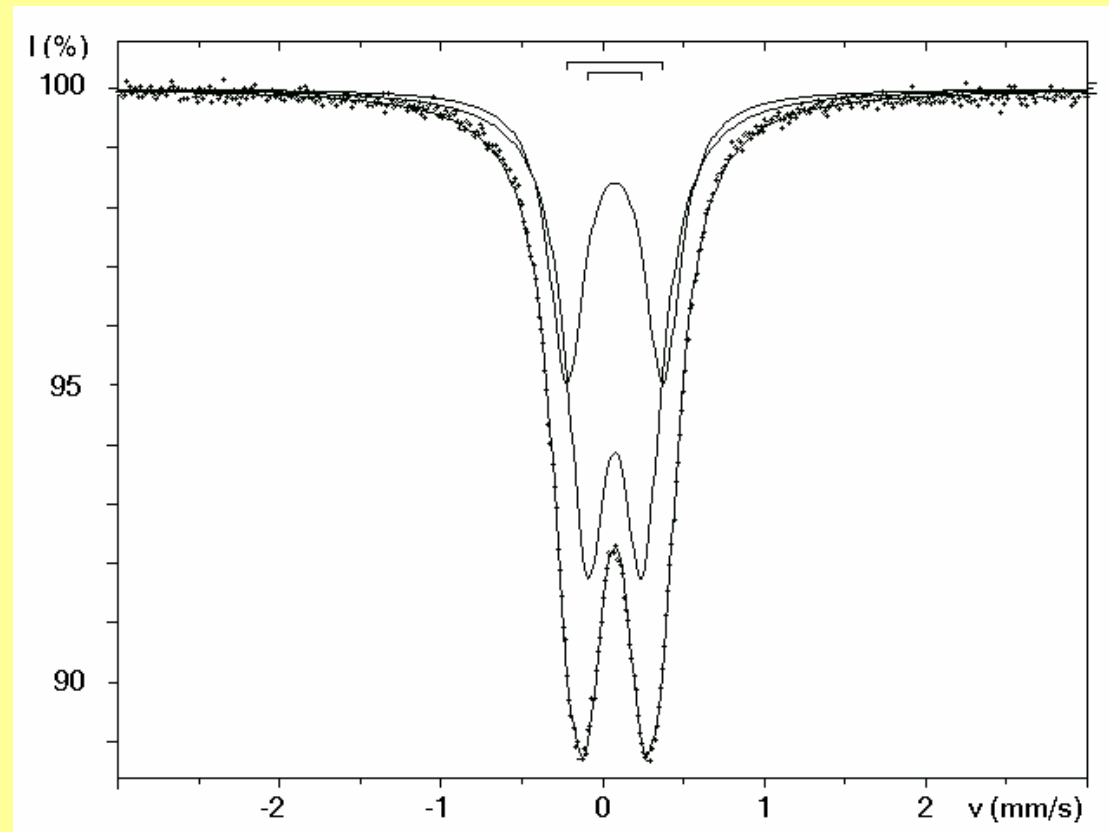
The linear heating of as-cast samples of both compositions exhibits distinct transformation stages.

Annealing temperature 473 K → no change of parameters of Mössbauer spectra.

According to TEM and XRD (Švec *et.al Mat. Sc. Eng. A375-377 (2004) 946-950*) evidence of presence of nanometer size of α -Al particles after first stage at 545 K.

Annealing at 623 K

Mössbauer spectra of $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$:



The value of Mössbauer parameters increased:

$$\begin{aligned}\bar{\delta}_1 &= 0.19 \text{ mm/s (0.18)} \\ \Delta_1 &= 0.59 \text{ mm/s (0.52)}\end{aligned}$$

$$\begin{aligned}\bar{\delta}_2 &= 0.19 \text{ mm/s (0.17)} \\ \Delta_2 &= 0.34 \text{ mm/s (0.29)}\end{aligned}$$



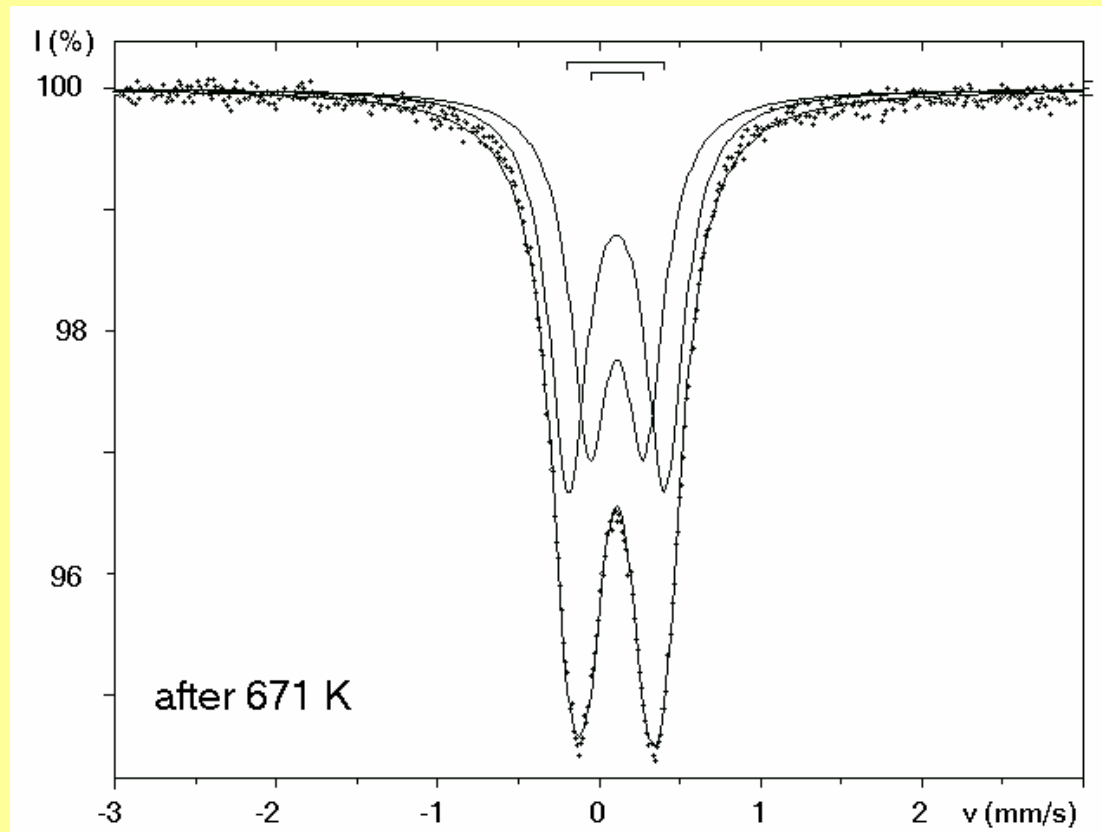
nanocrystalline
structure



1 – nanocrystalline Al (Fe), 2 – Al-Fe-Nb cluster

Annealing at 623 K

Mössbauer spectra of $\text{Al}_{94}\text{Fe}_2\text{V}_4$:



The value of Mössbauer parameters increased:

$$\begin{aligned}\delta_1 &= 0.21 \text{ mm/s (0.21)} \\ \Delta_1 &= 0.59 \text{ mm/s (0.59)}\end{aligned}$$

$$\begin{aligned}\delta_2 &= 0.22 \text{ mm/s (0.22)} \\ \Delta_2 &= 0.34 \text{ mm/s (0.33)}\end{aligned}$$



no change of parameters
comparing with as – cast sample

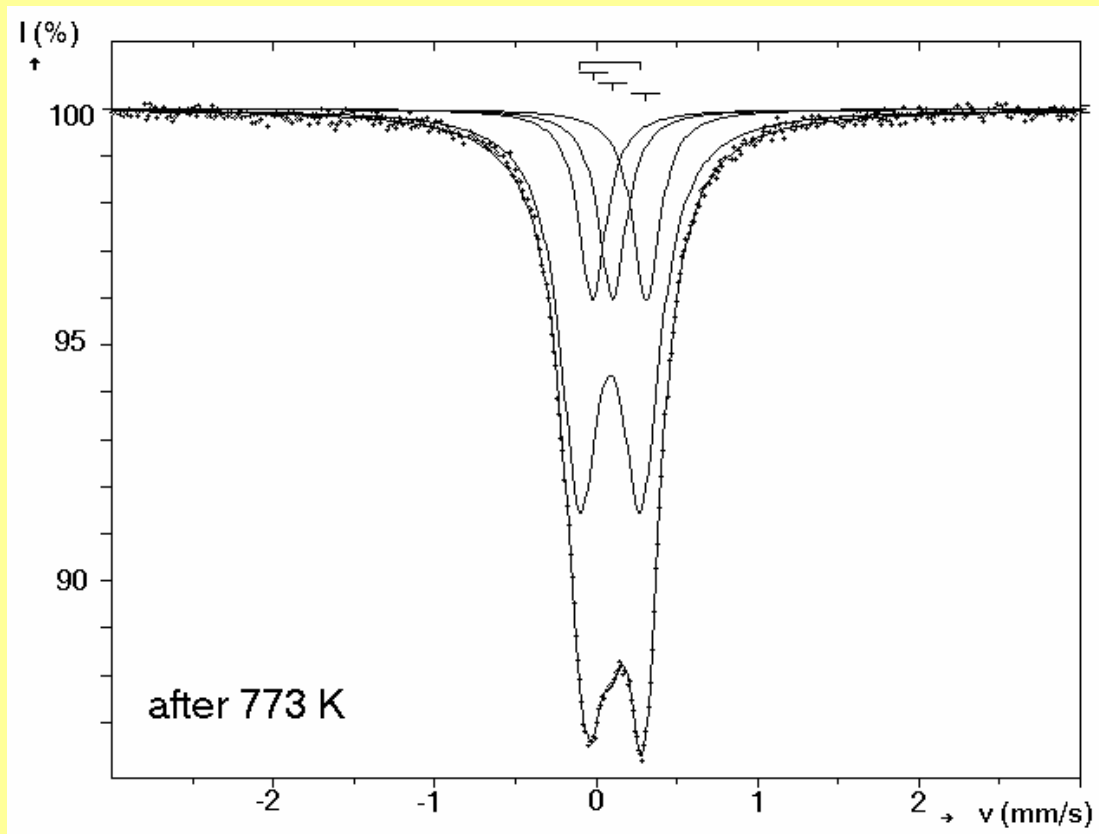


as – cast sample was already nanocrystalline

Annealing at 773 K

Next transformation stage is at 725 K. Above this temperature, intermetallic phases and α -Al are created.

Mössbauer spectra of $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$:



- 1) In the first step metastable phase Al_6Fe is created
- 2) This phase transforms to the intermetallic phase Al_3Fe ($\text{Al}_{13}\text{Fe}_4$)
- 3) Mössbauer spectrum is fitted by three singlets

(Forder et al., *Scripta Mat.* Vol.35, 1996, 1167)

(Nasu et al., *J.Physics C1*, 41, 1980, 385)

$\text{Al}_3\text{Fe} \dots \delta_4=0.09 \text{ mm/s}, \delta_5=0.21 \text{ mm/s}, \delta_6=0.42 \text{ mm/s}$

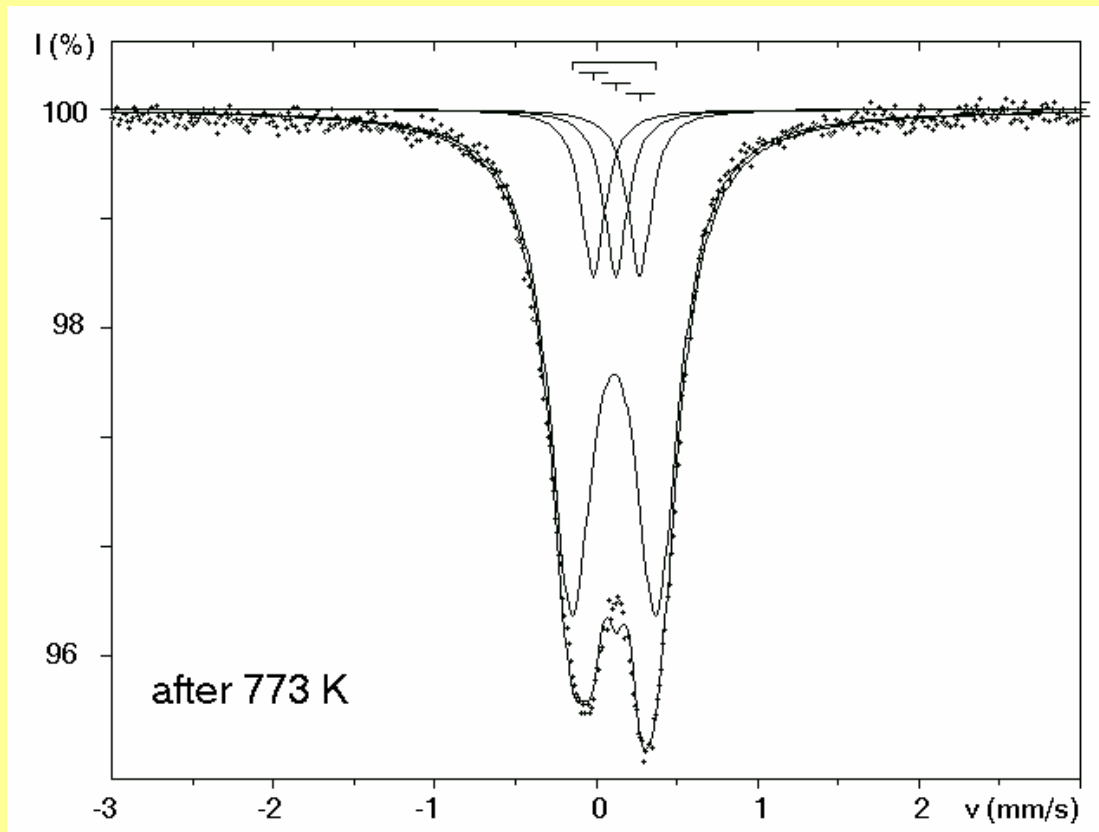
A=36%

$\text{Al}_6\text{Fe} \dots \delta_3=0.20 \text{ mm/s}, \Delta_3=0.38 \text{ mm/s}$ ($\delta = 0.23 \text{ mm/s}, \Delta = 0.30 \text{ mm/s}$)

A=64%

Annealing at 773 K

Mössbauer spectra of $\text{Al}_{94}\text{Fe}_2\text{V}_4$:



Al_3Fe → less amount than in previous sample

New doublet... polycrystalline aluminium containing Fe

?

Al_3Fe ... $\delta_4=0.09$ mm/s, $\delta_5=0.23$ mm/s, $\delta_6=0.38$ mm/s
New doublet... $\delta_3=0.22$ mm/s, $\Delta_3=0.52$ mm/s

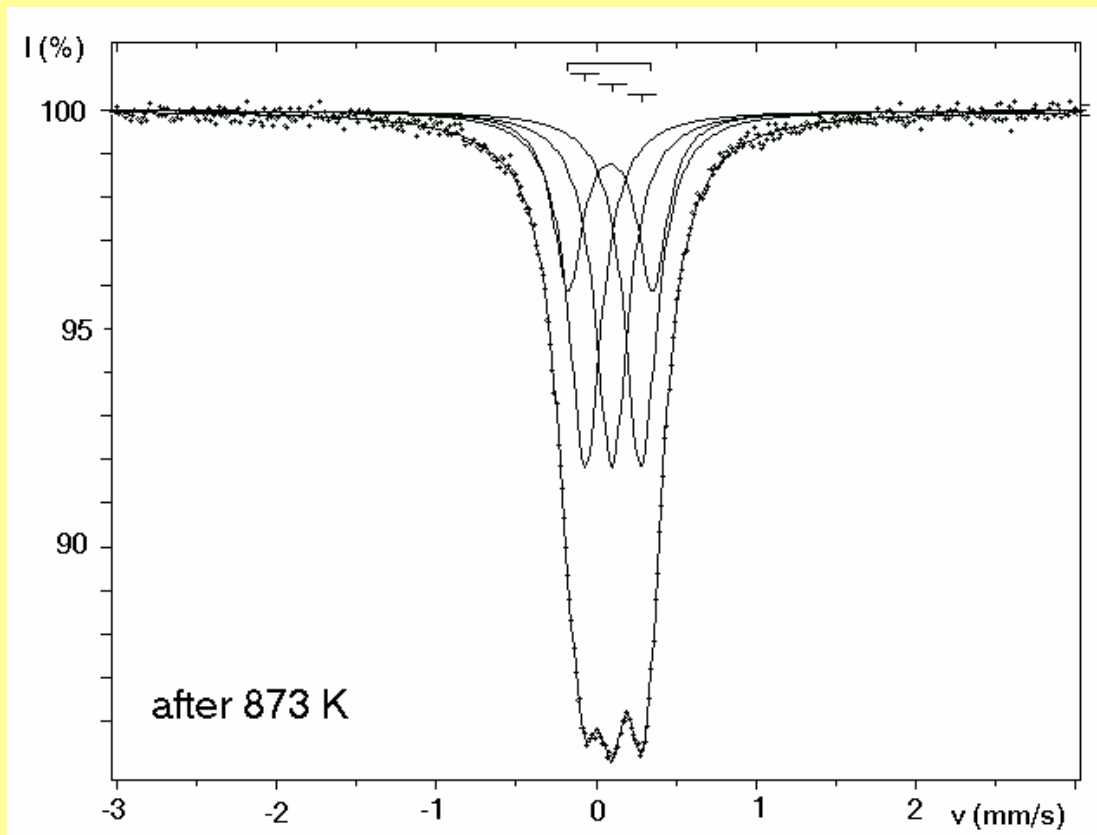
A=21%

A=79%

Annealing at 873 K

Next transformation stage is at 841 K. Above this temperature, intermetallic phases and polycrystalline aluminium are created.

Mössbauer spectra of $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$:



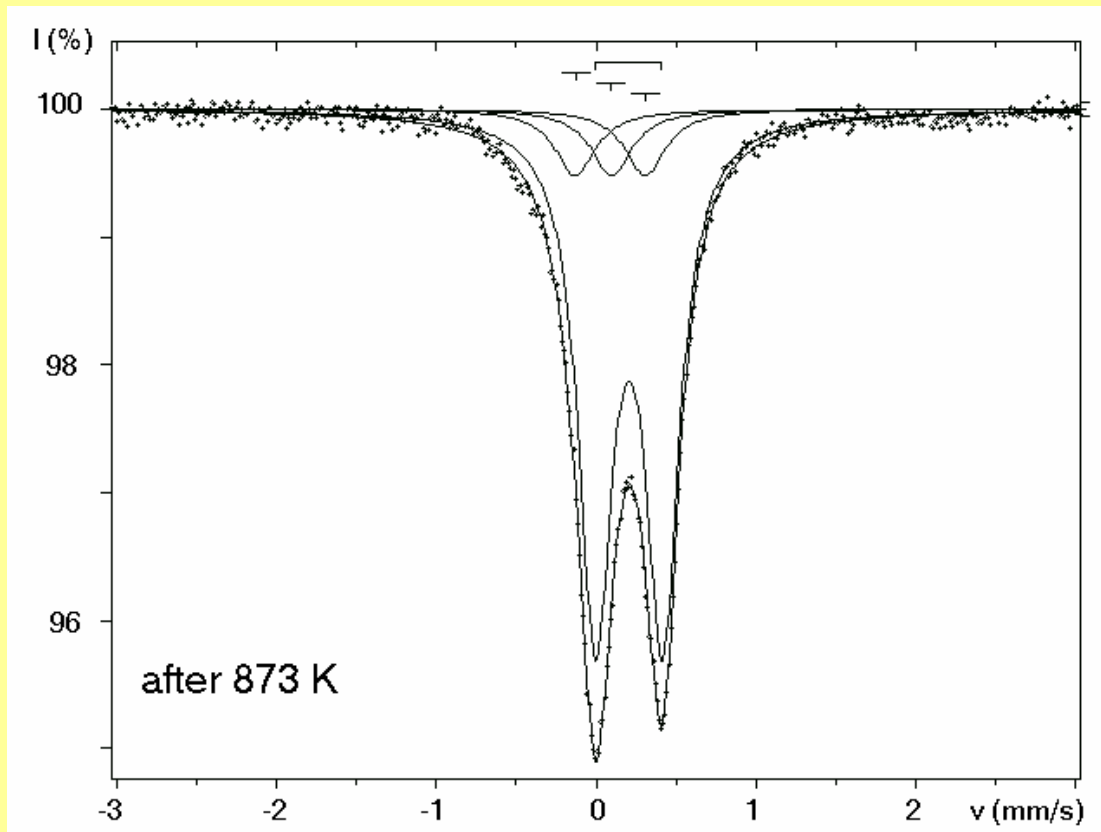
- 1) Dominant is Al_3Fe phase.
- 2) Doublet corresponds to the component containing iron in different sites as impurity in aluminium.

$\text{Al}_3\text{Fe} \dots \delta_4=0.04 \text{ mm/s}, \delta_5=0.21 \text{ mm/s}, \delta_6=0.39 \text{ mm/s}$
New doublet... $\delta_3=0.20 \text{ mm/s}, \Delta_3=0.53 \text{ mm/s}$

A=72%
A=28%

Annealing at 873 K

Mössbauer spectra of $\text{Al}_{94}\text{Fe}_2\text{V}_4$:



Mössbauer parameters of quadrupole splitting and isomer shift substantially changed.

We suppose:

Polycrystalline aluminium with the rest of intermetallic phases containing other constituent elements (Nb, V).

$\text{Al}_3\text{Fe}...$ $\delta_4=0.02$ mm/s, $\delta_5=0.21$ mm/s, $\delta_6=0.42$ mm/s
New doublet... $\delta_3=0.33$ mm/s, $\Delta_3=0.42$ mm/s

A=18%

A=82%

Conclusion

- 1) Amorphous and nanocrystalline state depends on additional constituent elements therefore
- 2) The alloy structure and its properties are modified by Fe, Nb and V elements
- 3) Crystallization process of sample $\text{Al}_{90}\text{Fe}_7\text{Nb}_3$ differs from the sample of $\text{Al}_{94}\text{Fe}_2\text{V}_4$
- 4) The alloy properties could be controlled by the crystallization process
- 5) The final properties of Al based alloys could be tailored by suitable combinations of constituent elements and by the annealing process