

Abstract

Diffusion couples experiments along with WDS analysis were used to measure the composition profiles of the formed phases of different systems. Boltzmann-Matano interface analysis has been performed on the obtained profiles to measure the atomic interdiffusivity of the system species. Several diffusion couples of Mg-Nd end-members were annealed at different times and temperatures to evaluate the linear growth of the diffusion layers with time. Moreover, the interdiffusion coefficients at the interface of the elements and intermetallic compounds, MgNd, Mg₃Nd and Mg₄₁Nd₅, were measured. One diffusion couple of Mn-Nd end-members annealed at 650°C for 30 days was enough to measure the interdiffusion coefficients at the interface of the three intermetallic compounds Mn₂Nd, Mn₂₃Nd₆ and Mn₁₇Nd₂. Two ternary diffusion couples composed of Mn₂₃Nd₆-Mg₃Nd and Mn₂₃Nd₆-MgNd end-members were annealed at 450°C for 20 and 5 days, respectively. The measured interdiffusion coefficients of the elements and intermetallic compounds were in the order of 10⁻¹³ to 10⁻¹⁵ cm²/sec; this can be referred to as the spatial resolution of the WDS spot analysis with 1-2µm displacement. Promising results on the binary systems were obtained, and more investigations are ongoing to evaluate the diffusivity of the species in the ternary system.

Methodology

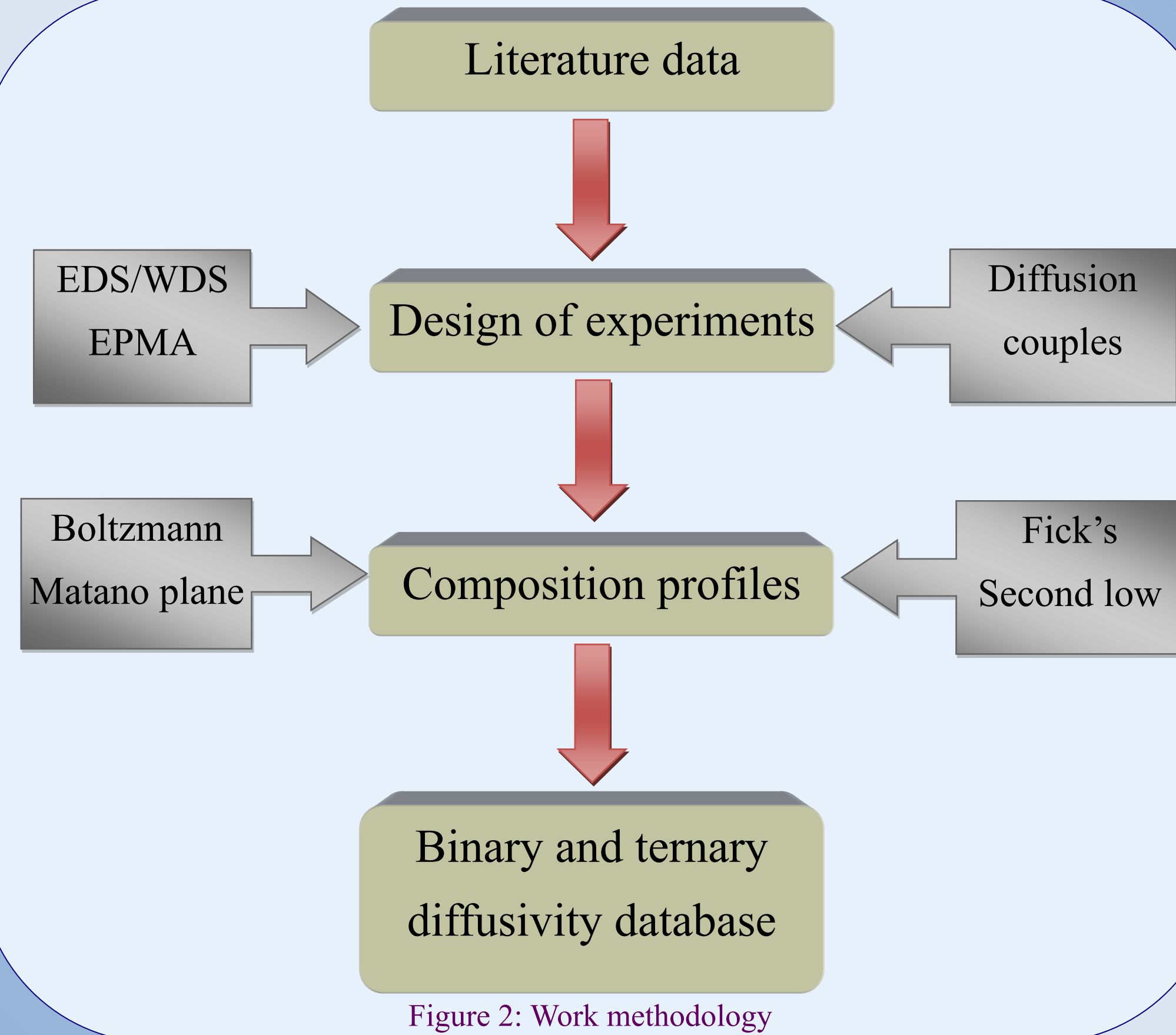


Figure 2: Work methodology

Introduction

Diffusivity measurements

$$\int_{c_1}^{c_2} xdc = 0$$

$$\bar{D}(c^{\phi}) = - \left(\int_{c_1}^{c_2} xdc \right) / (2t \frac{dc}{dx})$$

$\bar{D}(c)$ is the interdiffusivity at the composition C (cm²/sec), t is the annealing time (sec), dc/dx is the slope at the composition C (at.%/cm), and x is the layer thickness (cm)

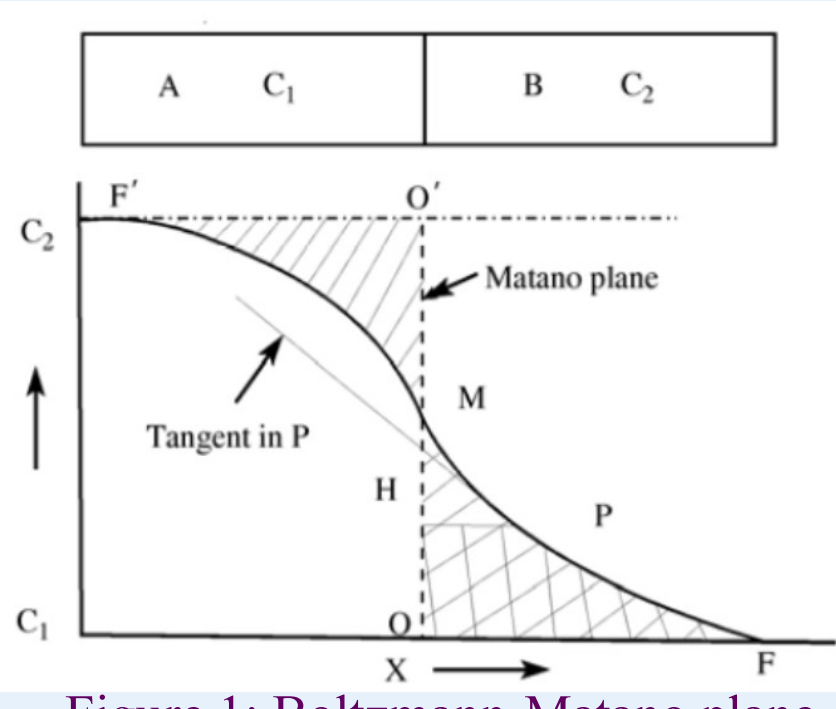


Figure 1: Boltzmann-Matano plane identification

Motivation

The microstructural information obtained in the alloy design processes are very important in determining the mechanical properties of alloys. Diffusion processes have a great influence on the alloys microstructures. Therefore, it is essential to understand the diffusivities of the system species. Very few attempts have been performed to measure the interdiffusion coefficients of the species in the Mg-Mn-Nd system. Although some information on the interdiffusion of Mg-Nd species is available in the literature, the results were inaccurate because the formation of Mg₄₁Nd₅ compound was not taken into consideration.

Objectives

The main objective of this work is to provide information on the interdiffusion coefficients at Boltzmann-Matano interface of the existing binary and ternary compounds in the Mg-Mn-Nd system. This information is necessary for practical applications such as solidification, precipitation, homogenization of alloys, recrystallization, grain boundary migration, creep-resistance enhancement, protective coatings, cladding, carburizing, nitriding, sintering, and joining processes.

Experimental Procedure

- Binary and ternary alloys were prepared initially from pure metals (99.98% Mg, 99.90% Mn, and 99.95%Nd).
- High melting temperature samples were prepared in an electrical arc-melting furnace; whereas, low-melting temperature alloys were prepared in an induction-furnace under argon.
- ICP was used to identify the chemical composition.
- Metals and alloys, used to make diffusion couples, were grinded down gradually up to 1200 grit size SiC sand papers. The grinded members then polished up to 1µm using alcohol-based diamond suspension.
- Metallic ring-clamps were used to attach the polished end-members to make solid-solid diffusion couples.
- Diffusion couples, wrapped up with Ta foil, were inserted in a hermetically sealed quartz tube under vacuum to be annealed at certain temperature and time.
- EDS/WDS were used to measure the composition profile of the diffused species.
- Boltzmann-Matano plane analysis were performed on the obtained profiles to measure the interdiffusion coefficient of the diffused species.



Figure 3: Induction-melting furnace



Figure 4: Clamped end-members



Figure 5: Samples inside a sealed quartz tube

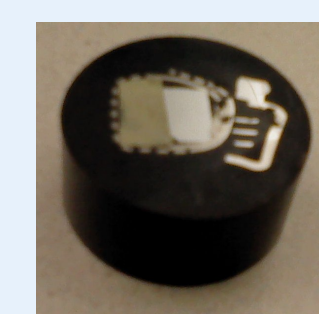


Figure 6: Mounted diffusion couple for WDS

Results and discussion

Diffusivity measurements of Mn-Nd system

- A diffusion couple made of pure Mn and Nd end-members was annealed at 650°C for 30 days.
- EPMA experiments used to measure the composition profile of the diffused species.
- Diffusivity measurements were performed on the profiles using Boltzmann-Matano calculations on the interfaces of the diffused phases.

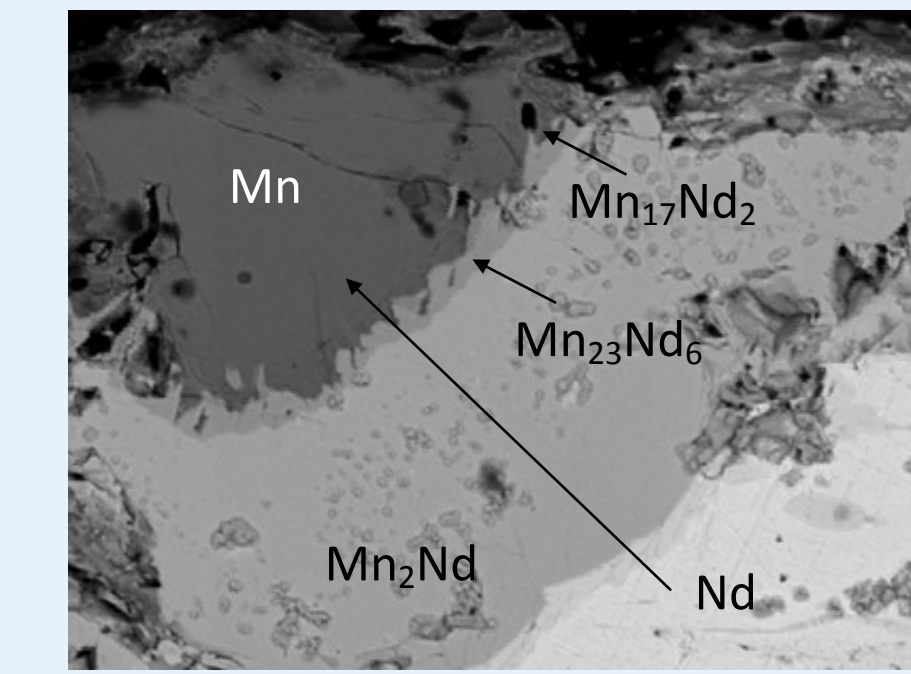


Figure 7: Mn-Nd diffusion couple

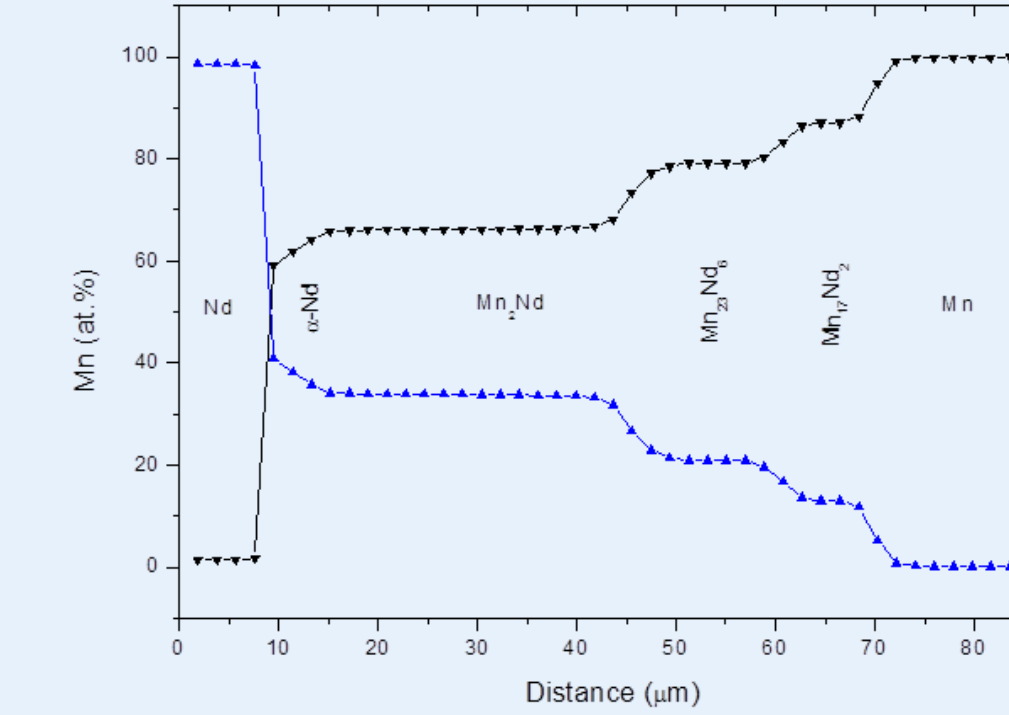


Figure 8: Measured composition profile

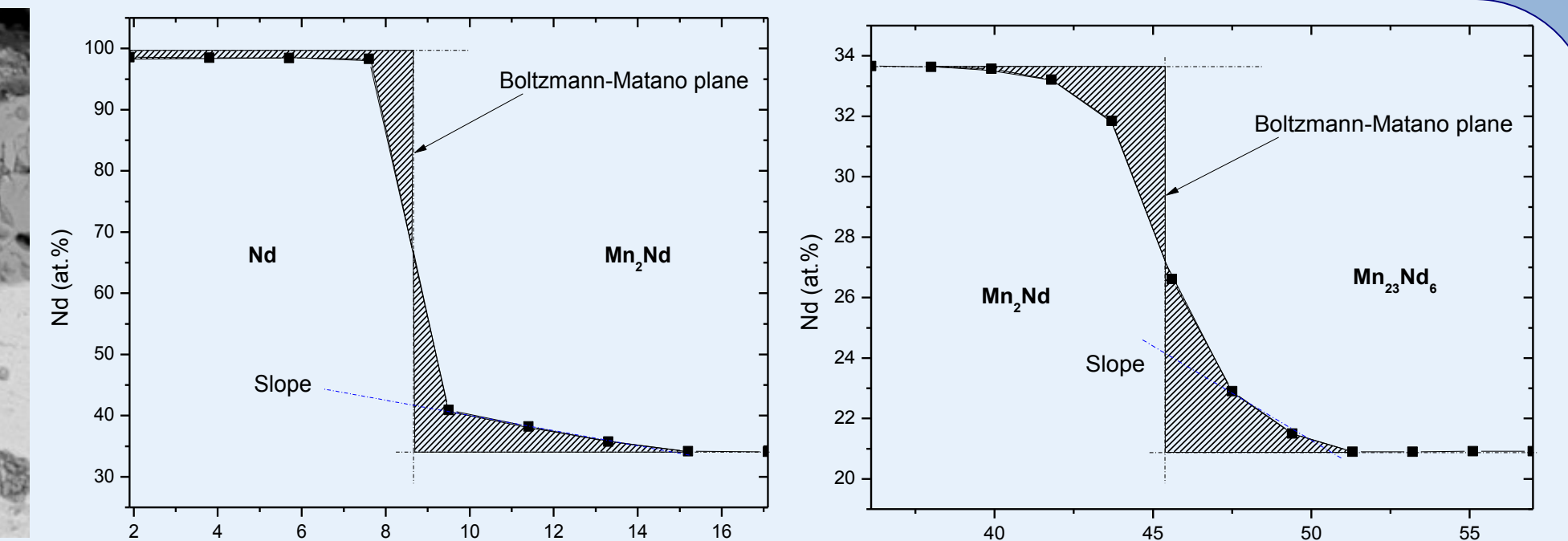


Figure 9 and 10: Plane position between Nd/Mn₂Nd and Mn₂Nd/Mn₂₃Nd₆ compounds

Table 1: Diffusivity measurements of Mn-Nd system species

Phase transition	dC/dx at.% Nd/cm	Area at.% Nd.cm	$\bar{D}(c)$ cm ² /sec
Nd→Mn ₂ Nd	-7431	0.025678	6.66×10 ⁻¹³
Mn ₂ Nd→Mn ₂₃ Nd ₆	-4073	0.003835	1.81×10 ⁻¹³
Mn ₂₃ Nd ₆ →Mn ₁₇ Nd ₂	-4470	0.00079	3.41×10 ⁻¹⁴
Mn ₁₇ Nd ₂ →Mn	-4207	0.001442	6.61×10 ⁻¹⁴

Diffusivity measurements of Mg-Nd system

- A diffusion couple made of pure Mg and Nd end-members was annealed at 400°C for 13 days.
- In contrast to the literature data, all phases were detected in the diffusion couple.
- Diffusivity measurements were comparable to the available literature data except for Mg₃Nd/Mg₄₁Nd₅ and Mg₄₁Nd₅/Mg interfaces; since Mg₄₁Nd₅ was not detected.

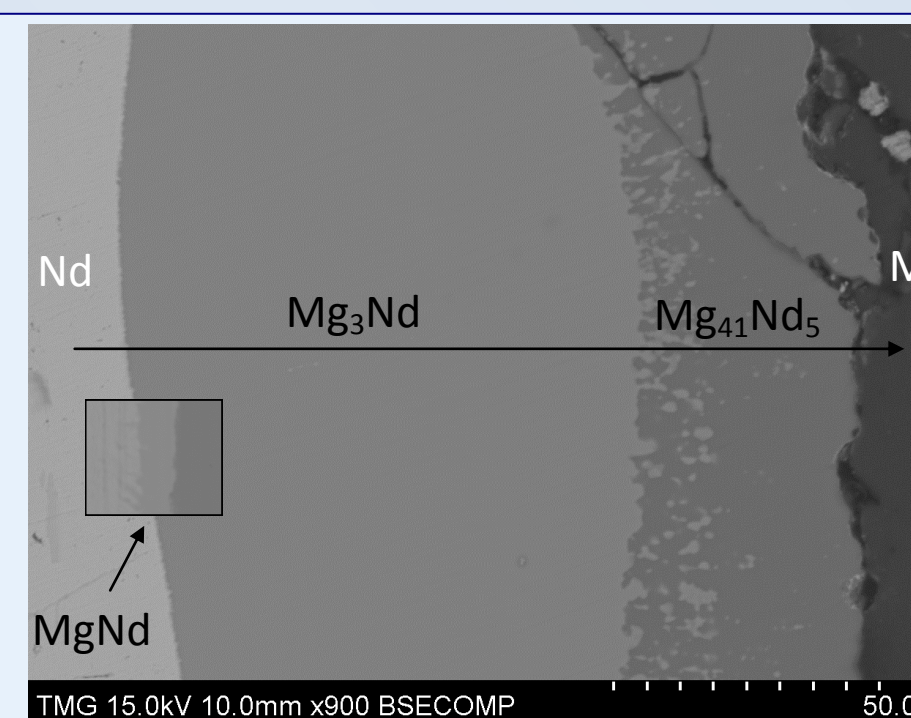


Figure 11: Mg-Nd diffusion couple

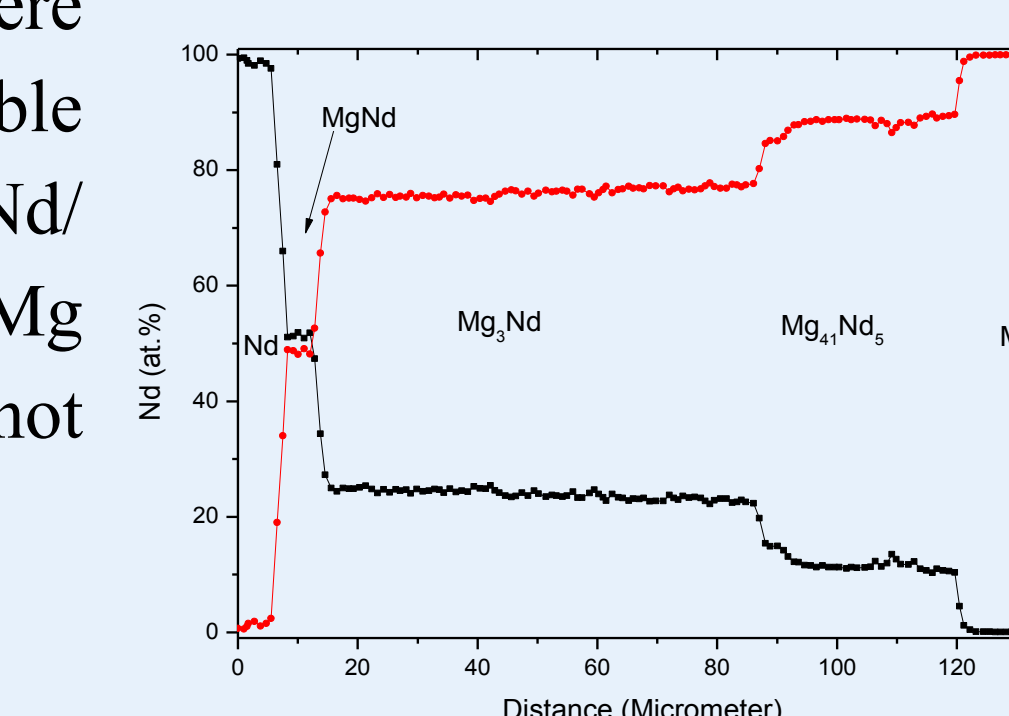


Figure 12: Measured composition profile

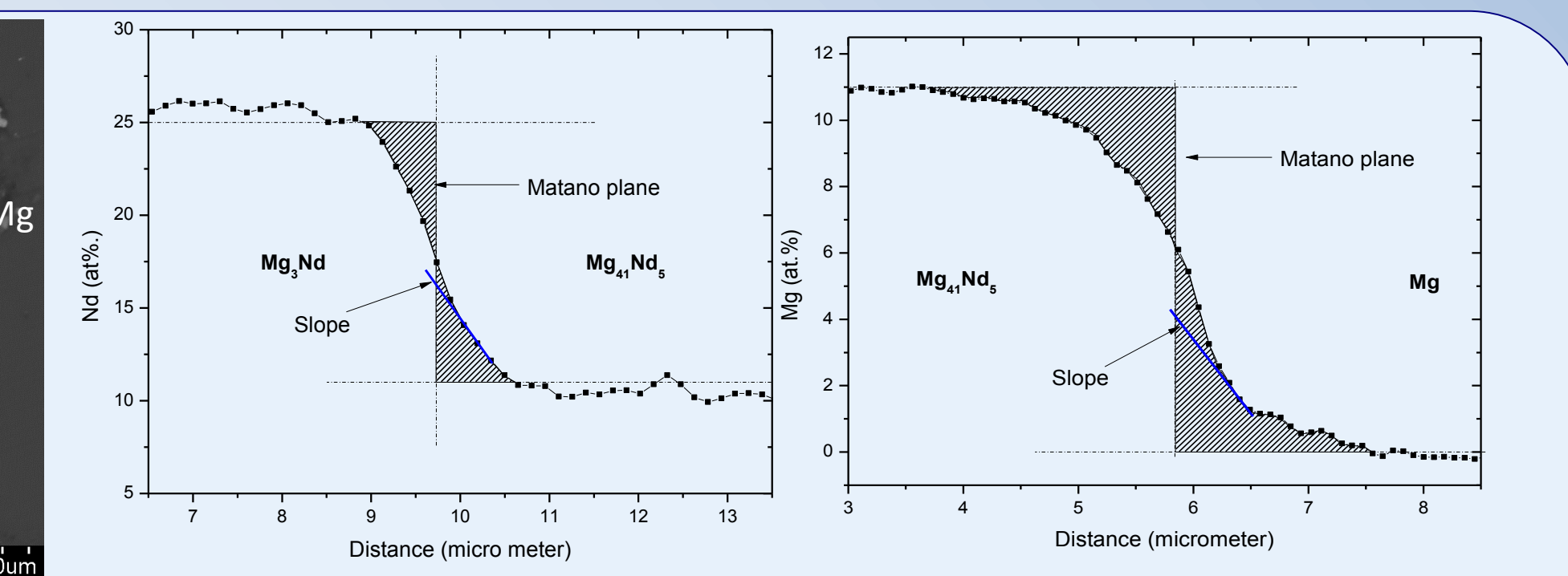


Figure 13&14: Plane position between Mg₃Nd/Mg₄₁Nd₅ and Mg₄₁Nd₅/Mg compounds

Table 2: Diffusivity measurements of Mg-Nd system species

Phase transition	dC/dx at.% Nd/cm	Area at.% Nd.cm	$\bar{D}(c)$ cm ² /sec
Nd→MgNd	140480	0.002409	7.63×10 ⁻¹⁵
MgNd→Mg ₃ Nd	35620	0.001092	1.36×10 ⁻¹⁴
Mg ₃ Nd→Mg ₄₁ Nd ₅	5385	0.00175	1.44×10 ⁻¹³
Mg ₄₁ Nd ₅ →Mg	10590	0.000475	1.99×10 ⁻¹⁴

Mg-Mn-Nd diffusion couples results

- Two diffusion couples made of Mn₂₃Nd₆-Mg₃Nd and Mn₂₃Nd₆-MgNd end-members were annealed at 450°C for 20 and 5 days, respectively.
- Alloys composed of Mn-Nd elements have high tendency to dissolve Mg species as shown in Figure 15.
- The results obtained by the two ternary diffusion couples showed not intersected diffusion paths..

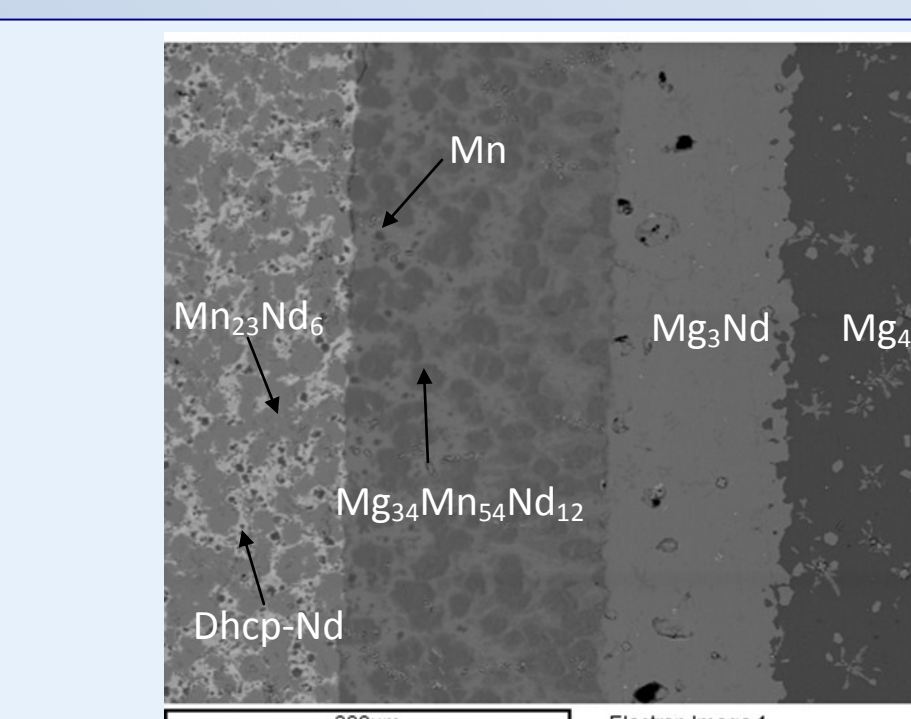


Figure 15: Mn₂₃Nd₆/Mg₄₁Nd₅ diffusion couple

- The tie line from Mn passing through Mg₃Nd towards Mg₄₁Nd₅ was confirmed by the results obtained from the ternary diffusion couple.

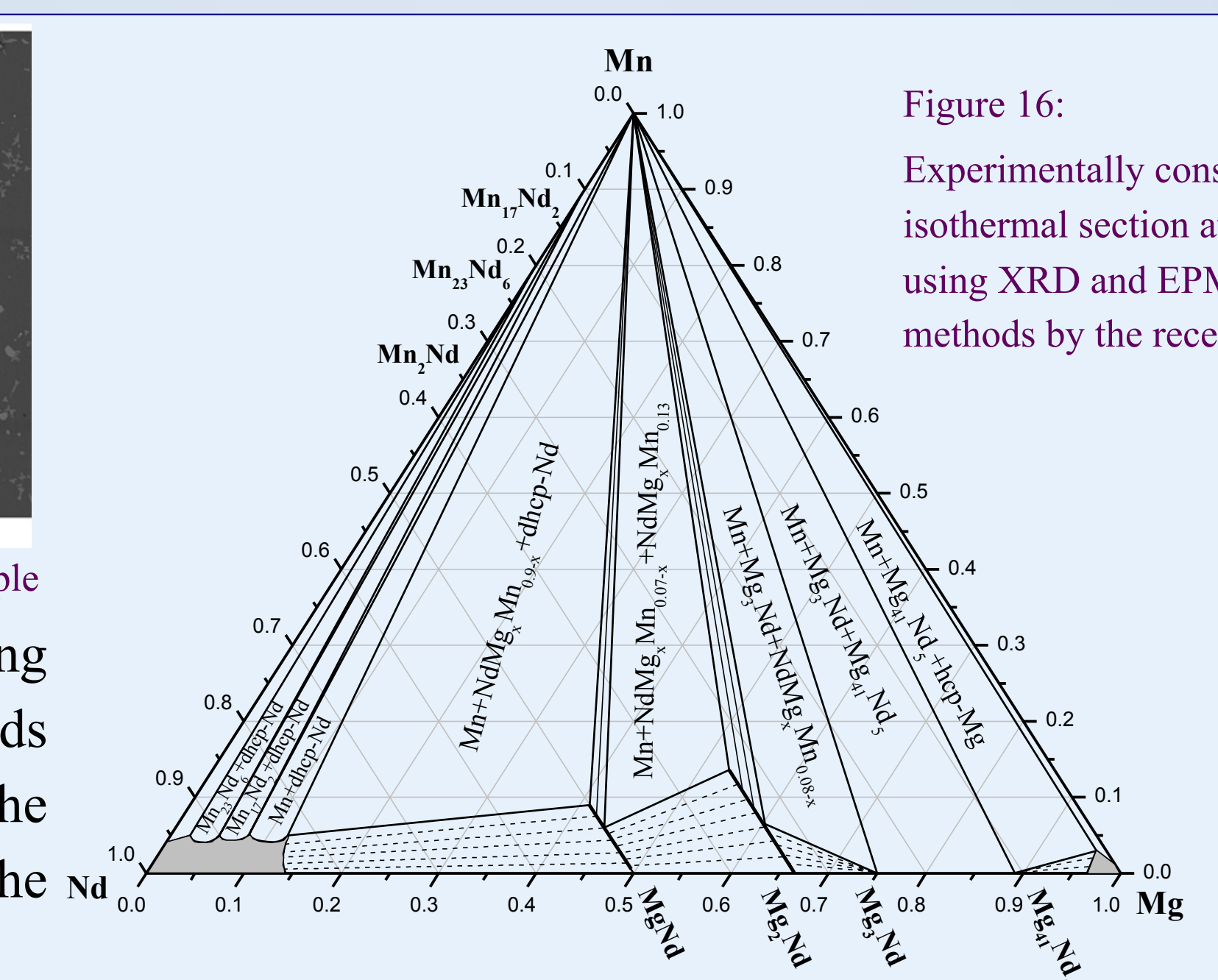


Figure 16: Experimentally constructed isothermal section at 450°C using XRD and EPMA/WDS methods by the recent authors.

Conclusions

- Binary diffusivities were measured and found comparable to the available data in the literature. The diffusivity values were found in the order of 10⁻¹³ to 10⁻¹⁵; this can be referred to the spatial WDS displacement of 1-2µm.
- The diffusivity measurements of the ternary Mg-Mn-Nd system will be evaluated by making ternary diffusion couples with intersected diffusion paths. This will help in reducing the number of the boundary conditions during the ternary diffusivity identification.

Acknowledgments

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