

Fundamental investigation of the technological research on the reaction sintering of MgAlON at 1500 °C from Al₂O₃, MgO and AlN

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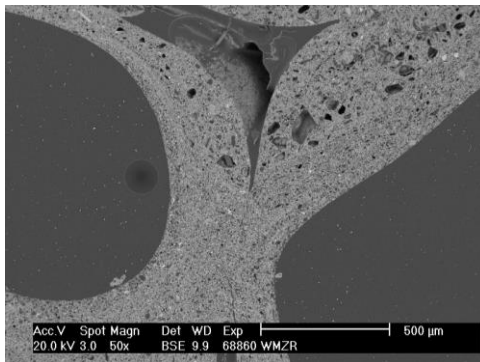


Table of contents

1. Aim of the fundamental investigation of MgAlON
2. Starting Composition (Al_2O_3 , MgO, AlN)
3. Theoretical and thermodynamic consideration
4. Summary of Results
5. Conclusion
6. Acknowledgment

MDPI “Crystals”

*Special Issue “High-Tech Metals – Overview,
Microstructure, Properties and Recyclability”*

1. Aim of the Fundamental Investigation of MgAlON

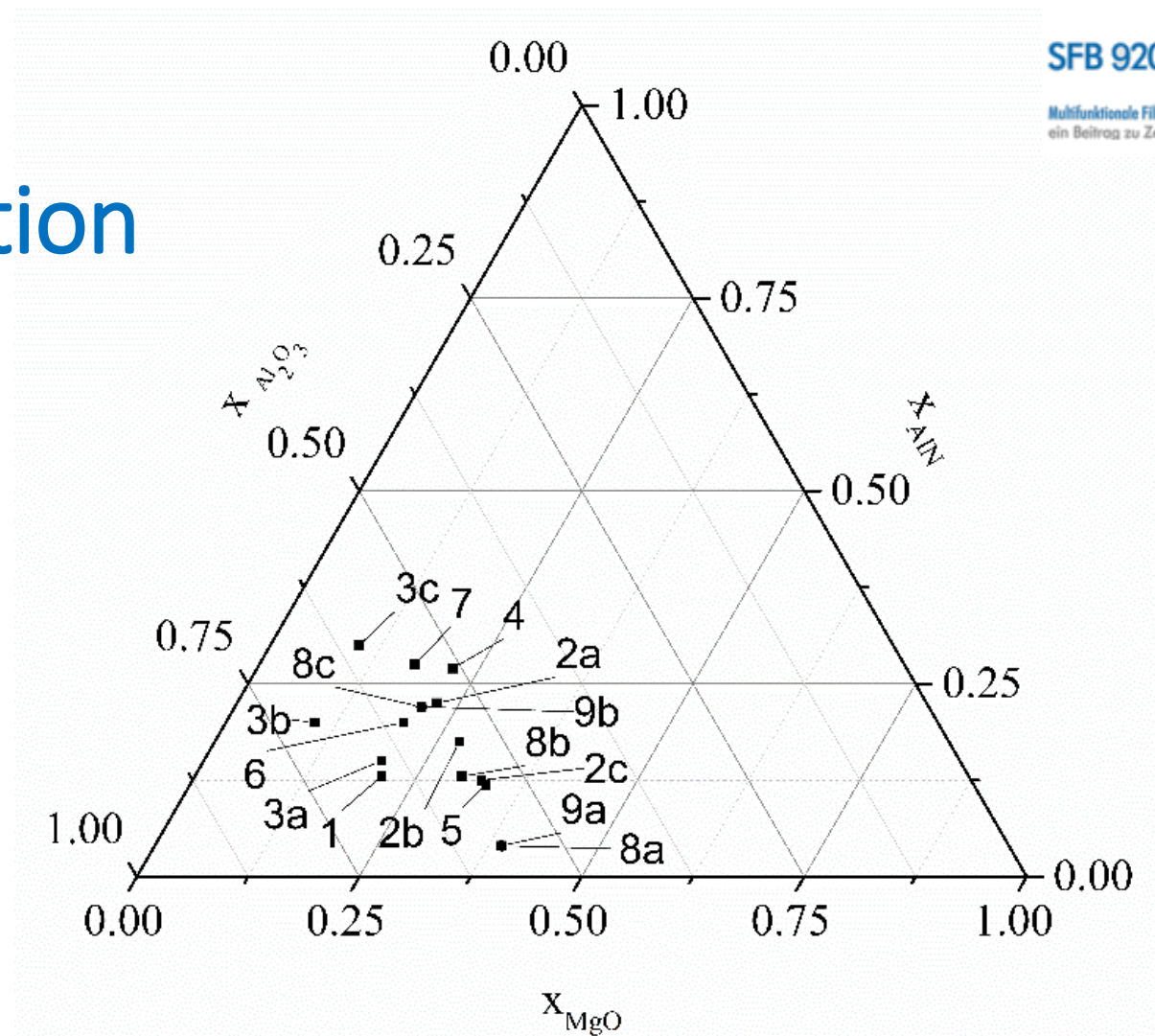
Ceramic Foam Filters => Purification of metal melts,
especially for Magnesium as a light-weight material

Coating Material for Ceramic Foam Filters = MgAlON

Investigation of the technological synthesis of MgAlON at 1500°C in
nitrogen atmosphere

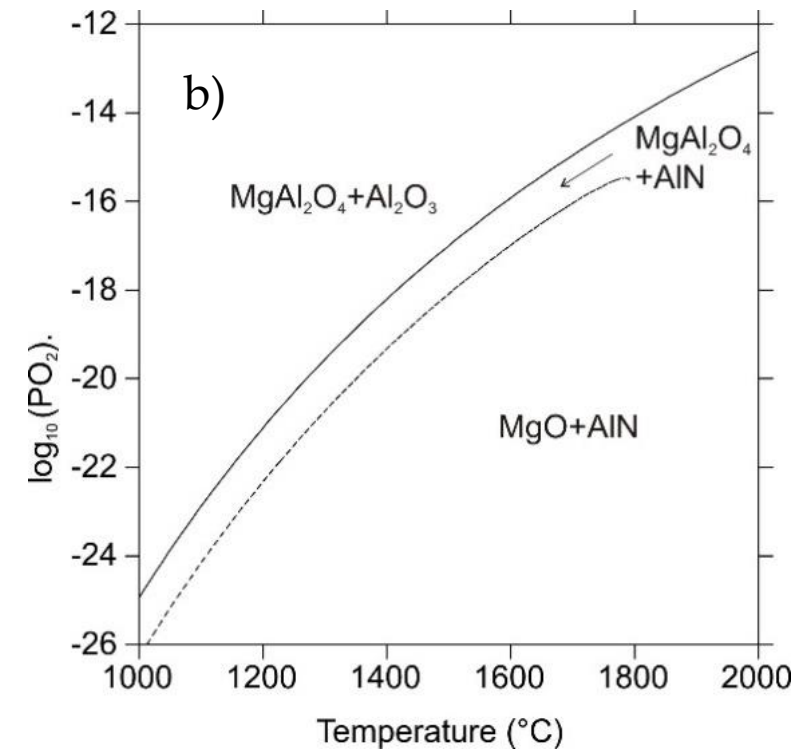
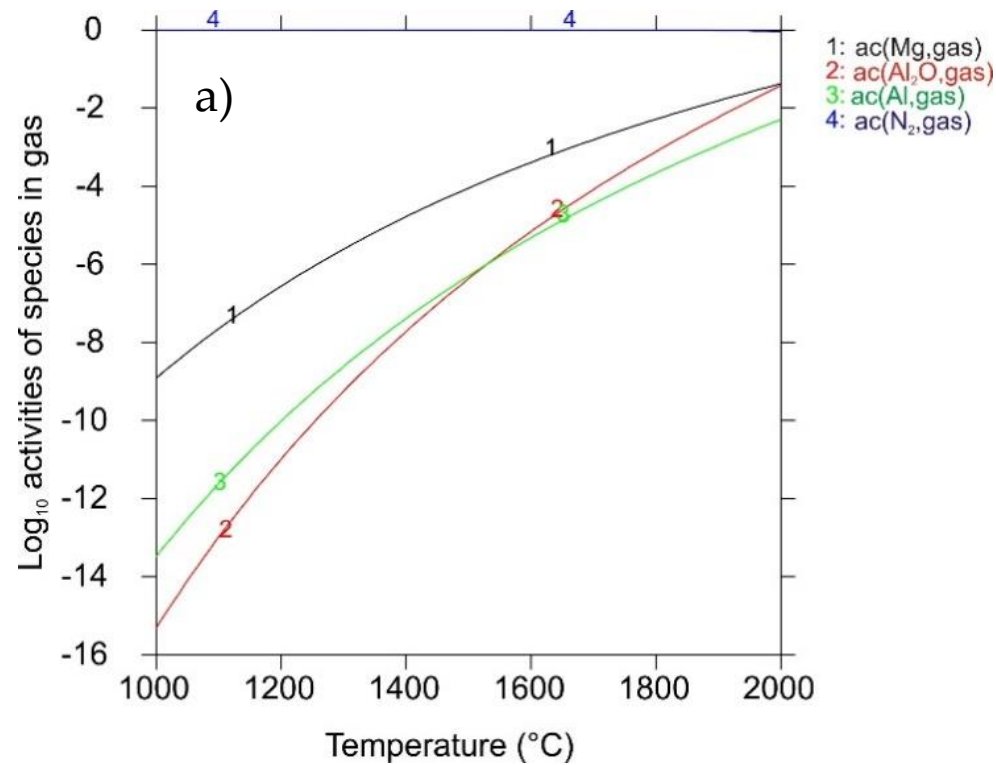
2. Starting Composition

- Samples in the ternary system Al_2O_3 - MgO - AlN



Schramm, A., Thümmeler, M., Fabrichnaya, O., Brehm, S., Kraus, J., Kortus, J., Rafaja, D., Scharf, C., Aneziris, C. G.: Reaction Sintering of MgAlON at 1500 °C from Al_2O_3 , MgO and AlN and Its Wettability by AlSi_7Mg , Crystals 2022, 12, 654. <https://doi.org/10.3390/cryst12050654>

3. Theoretical and thermodynamic consideration

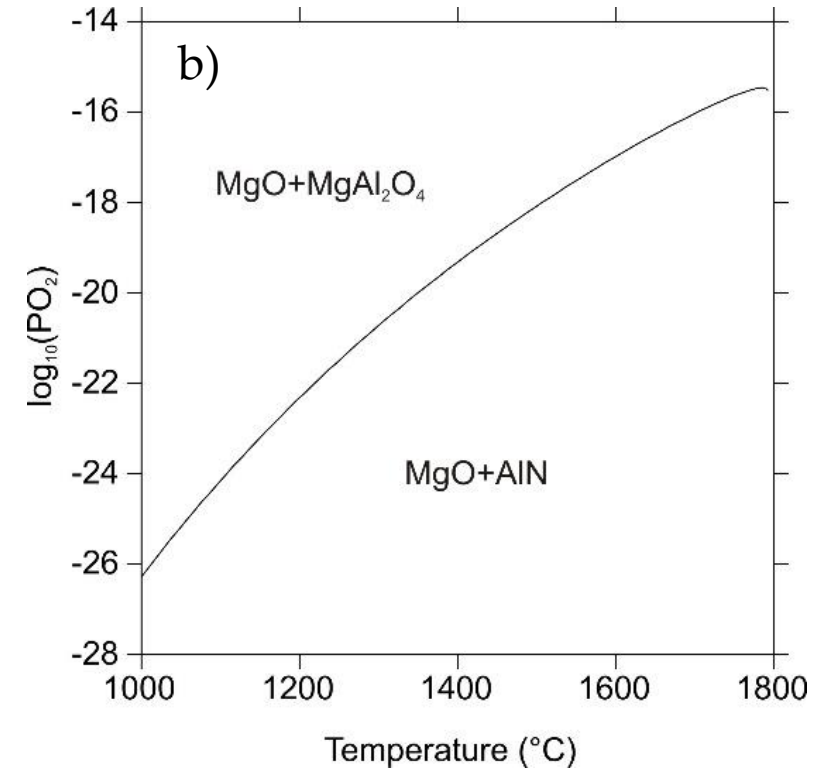
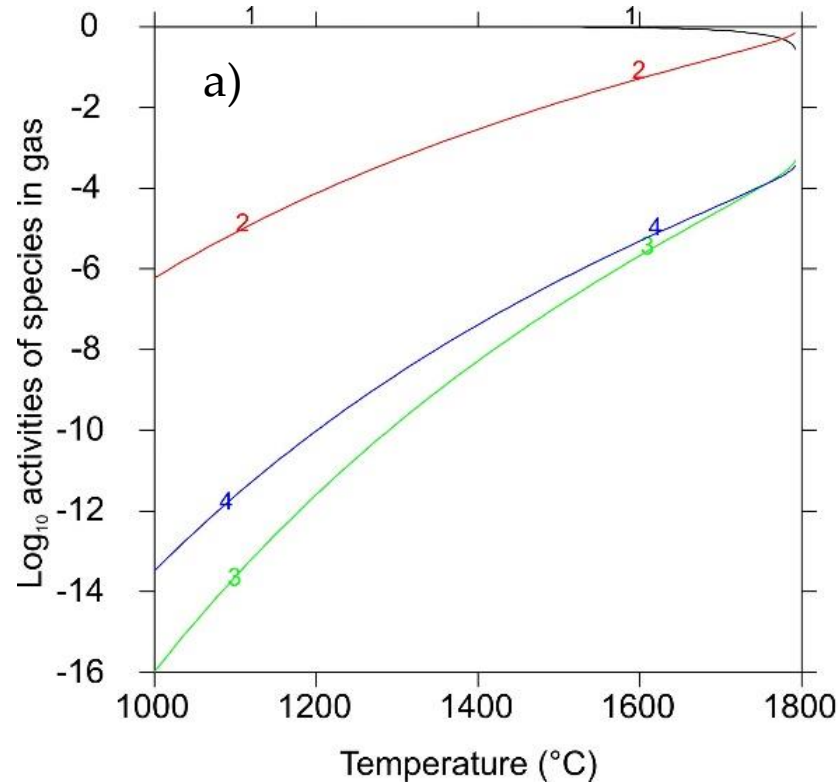


Equilibrium of MgAl_2O_4 with Al_2O_3 and AlN in presence of gas as a function of temperature:

(a) activities of gas species;

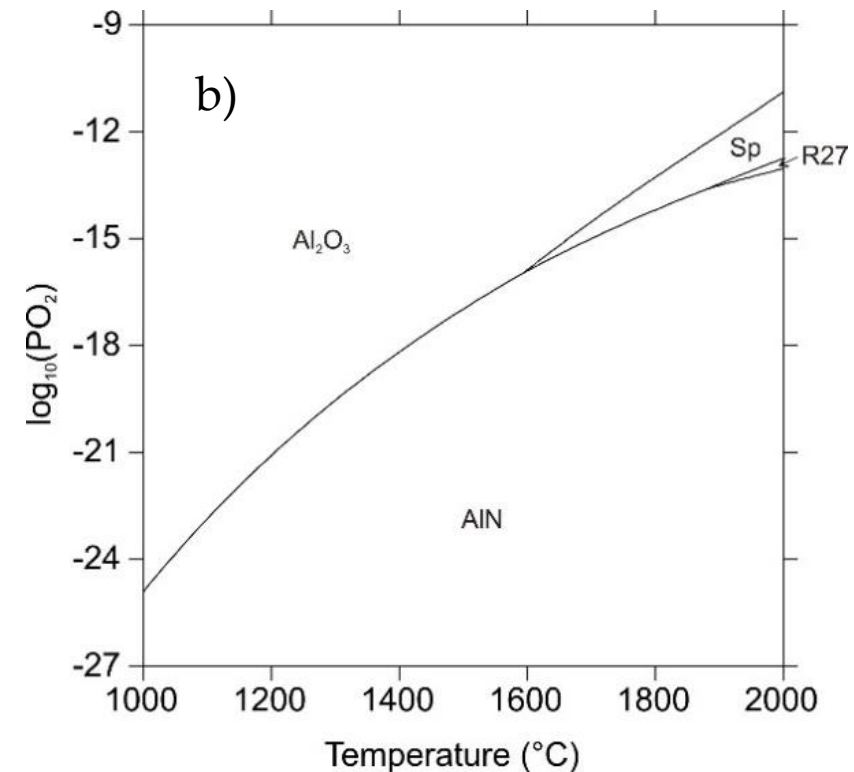
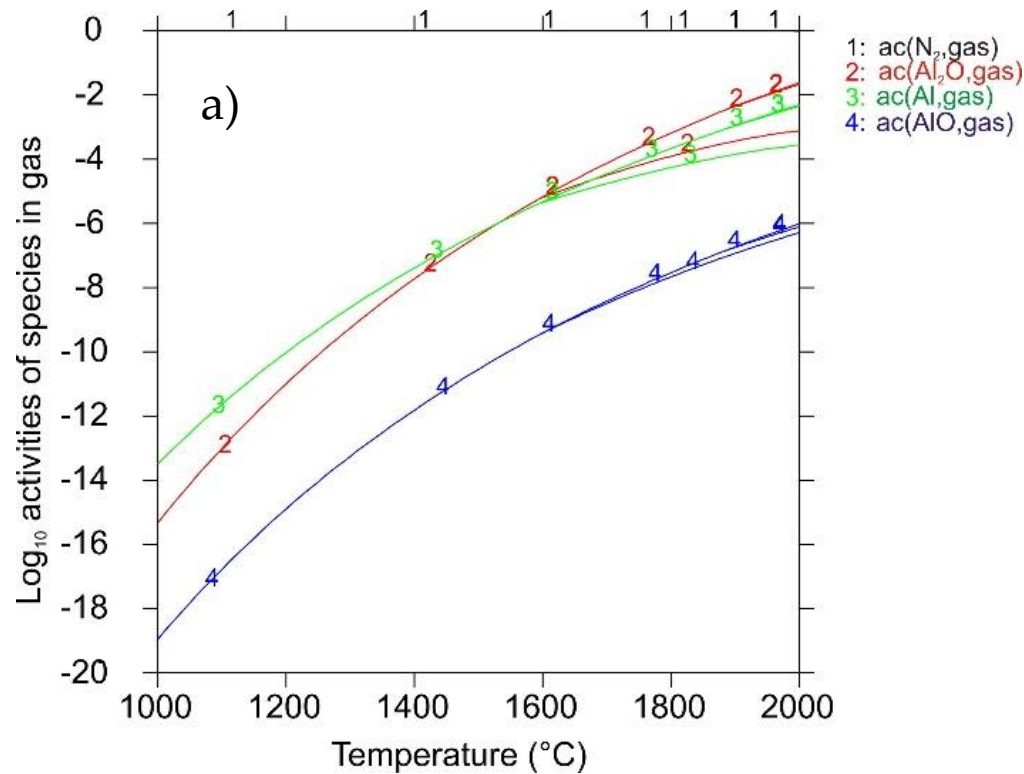
(b) partial pressure of oxygen, the dashed line shows the stability limit of MgAl_2O_4 .

3. Theoretical and thermodynamic consideration



Equilibrium of MgAl₂O₄ with MgO and AlN in presence of gas as a function of temperature: (a) activities of gas species; (b) partial pressure of oxygen.

3. Theoretical and thermodynamic consideration

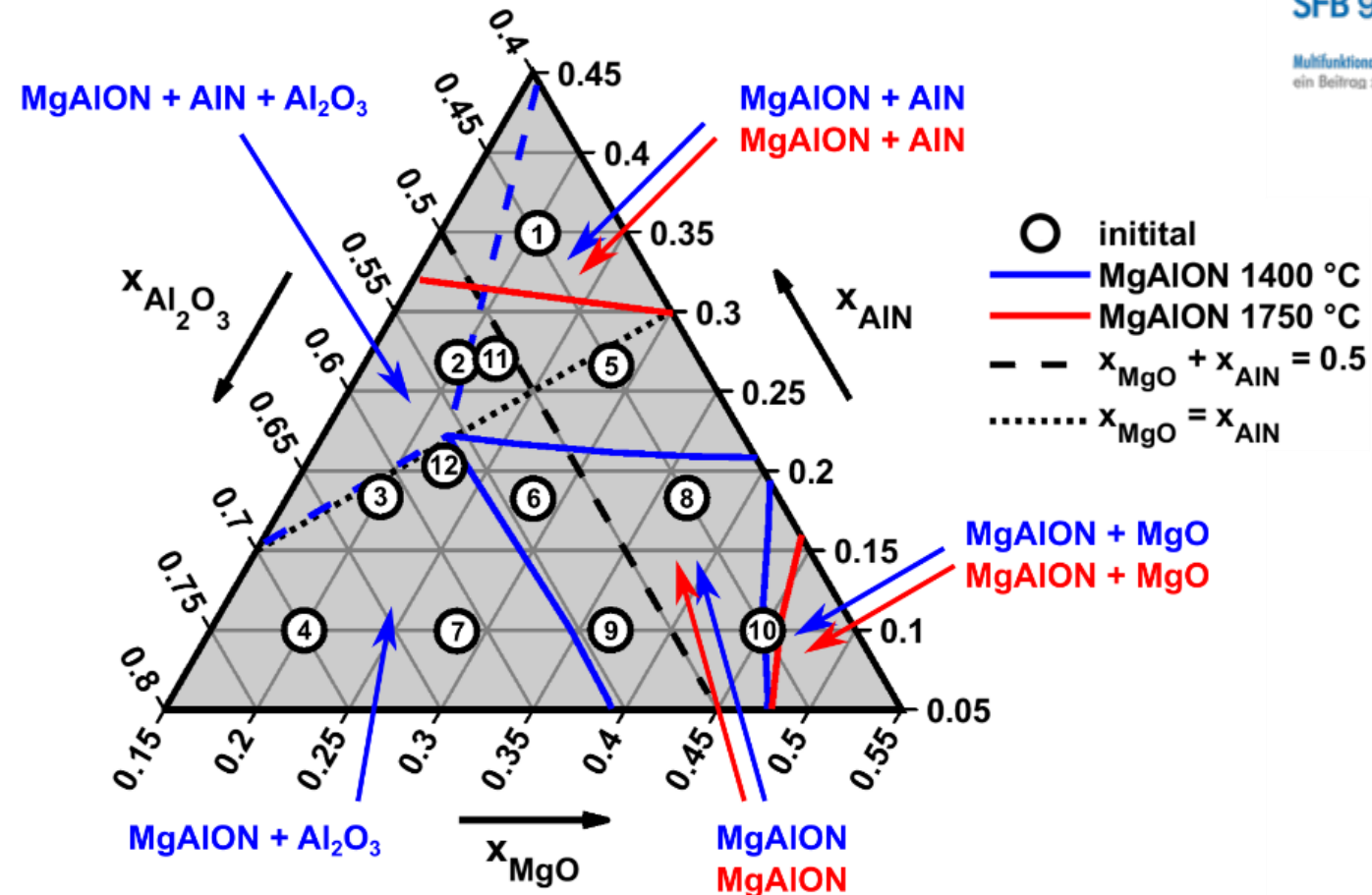


Phase equilibria in the Al_2O_3 -AlN system Al_2O_3+AlN , $Al_2O_3+Spinel AlON$, $Spinel AlON+AlN$, $R27+Spinel AlON$ and $R27+AlN$ in presence of gas: (a) temperature dependence of the activities of gas species; (b) temperature dependence of the partial pressure of oxygen.

3. Theoretical and thermodynamic consideration

Standard Gibbs energies of reaction $\Delta_r G^\circ$ at 1500 °C and 1 bar, calculated using PBE-DFT.

Reaction No.	Chemical Equation	$\Delta_r G^\circ_{\text{DFT}}$ (kJ mol ⁻¹)	$\Delta_r G^\circ_{\text{ref1}}$ (kJ mol ⁻¹)
1	$\text{MgO}_{(s)} \rightarrow \text{Mg}_{(g)} + \frac{1}{2} \text{O}_{2(g)}$	325.4	366.3
2	$\text{MgAl}_2\text{O}_{4(s)} \rightarrow \text{Al}_2\text{O}_{3(s)} + \text{Mg}_{(g)} + \frac{1}{2} \text{O}_{2(g)}$	351.6	408.3
3	$\text{Al}_2\text{O}_{3(s)} + 4 \text{AlN}_{(s)} \rightarrow 3 \text{Al}_2\text{O}_{(g)} + 2 \text{N}_{2(g)}$	681.9	793.4
4	$\text{MgAl}_2\text{O}_{4(s)} + 4 \text{AlN}_{(s)} \rightarrow 3 \text{Al}_2\text{O}_{(g)} + \text{Mg}_{(g)} + \frac{1}{2} \text{O}_{2(g)} + 2 \text{N}_{2(g)}$	1033.4	1201.7
5	$\text{AlN}_{(s)} \rightarrow \text{Al}_{(g)} + \frac{1}{2} \text{N}_{2(g)}$	247.0	230.8
6	$2 \text{Al}_{(g)} + \frac{1}{2} \text{O}_{2(g)} \rightarrow \text{Al}_2\text{O}_{(g)}$	-529.3	-485.5



Compositions of the initial powder mixtures used, shown in a simplified phase diagram for the ternary system MgO-AlN-Al₂O₃. The homogeneity ranges of MgAlON and the adjacent multiphase regions for 1400 °C and 1750 °C were estimated according to the phase diagrams proposed by Willems [Preparation and Properties of Translucent Gamma-Aluminium Oxynitride. PhD Thesis, Department of Chemical Engineering and Chemistry, Technische Universiteit Eindhoven, The Netherlands, 1992].

4. Summary

- Pressureless reactive sintering of Al_2O_3 -AlN-MgO mixtures at a sintering temperature of 1500 °C with a duration of 3 hours does not result in a near-equilibrium state and yields an average conversion degree to MgAlON of only 76 wt%.
- Reactive sintering of the same mixtures at 1500 °C for 6 hours results in a near-equilibrium state, reaching an average conversion degree to MgAlON of 91.9 wt%.
- The highest conversion degrees of 99 wt% of MgAlON were reached in the MgO-rich corner of the quasi-ternary MgO-AlN- Al_2O_3 system with Al_2O_3 :AlN ratios of around 20:6.
- At 1500 °C, MgAlON forms from MgAl_2O_4 , which is a direct reaction product of MgO and Al_2O_3 , and the subsequent, simultaneous dissolution of Al_2O_3 and AlN in the spinel structure. During this process, MgO is fully consumed after the first 3 hours; the loss of magnesium due to evaporation or parasitic reactions is insignificant.
- The relative amount of nitrogen in the MgAlON phase of the samples with the lowest and highest conversion degree after 6 hours of reactive sintering was measured to be 1.5 wt% for both samples.

5. Conclusion

Reaction sintering of MgAlON delivers suitable coated ceramic foam filter material for the refining of light metal melts (Mg)

6. Acknowledgement

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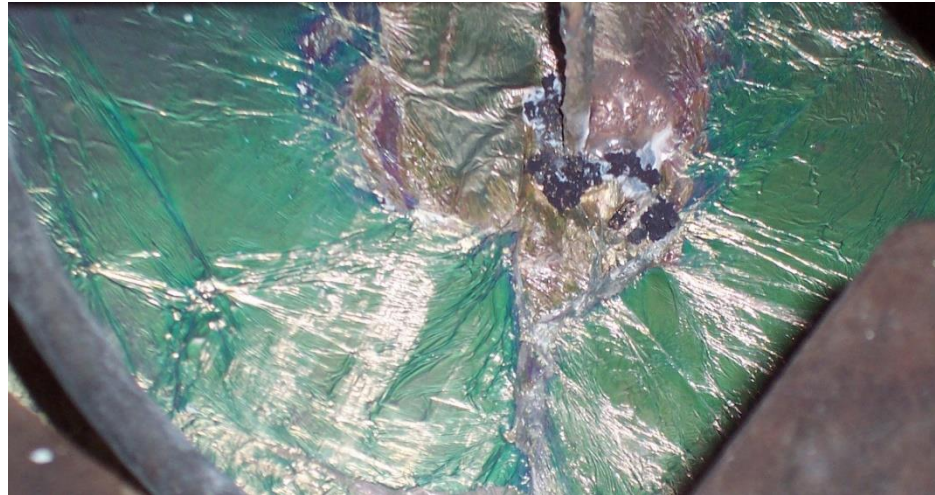
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