The Influence of Dopant Species on Alumina Grain Boundary Mobility

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Introduction & Motivation

- The mechanical and functional properties of polycrystalline ceramics materials strongly depend on the grain size, which is controlled by grain growth during powder densification in the sintering process[1].
- The extent of grain growth is directly related to grain boundary (GB) velocity, which depends on GB energy and GB mobility[2]. Therefore, GB mobility is one of the key parameters that dominates the properties of polycrystalline materials[1,3].
- Chemical segregation to GBs can affect the GB velocity. Dopants and/or impurities can significantly change the GB mobility of materials[1,3-5]. The segregated solutes can then induce solute drag or solute acceleration, changing the grain boundary mobility and the microstructure evolves[1,3]
- In this ongoing study, the influence of Fe as a solute on the GB mobility of alumina is studied.

Methodology

Powder preparation

① α-Al_2O_3 powder (purity 99.99%)

$Fe(NO)_3 \cdot 9H_2O$ (purity 99.99%)

② RTP Al_2O_3 powder(with 132ppm Mg)

RTP= Ready to Press

Green bodies preparation

green body by slip casting — undoped

green body by slip casting — Fe-doped, Fe-doped, Fe-doped

Fe&Mg -doped

green body by uniaxial pressing (~27MPa) — Mg-doped

Sintering

1600°C, 2h, in air to achieve fully dense

Fe concentration by SEM-WDS

Atual dopants' concentration

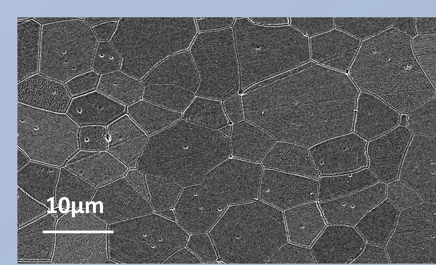
$$C_{spec} = \frac{I_{spec}}{I_{std}} \cdot g_{ZAF} \cdot C_{std}$$

undoped
0.18wt%Fe
0.71wt%Fe
1.26wt%Fe
0.41wt%Fe +132ppm Mg
132ppm Mg

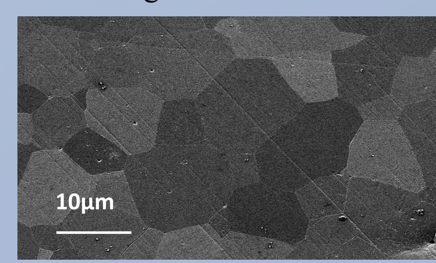
C_{spec} - concentration of element.
I_{spec} - intensity of characteristic X-ray radiation from the specimen.
I_{std} - intensity measured from a selected standard FeS_2.
g_{ZAF} - correction factor for atomic number, absorption, and fluorescence.
C_{std} - element concentration of standard.

Grain growth

Annealing at 1600°C for 1min, 1h, 2h, 4h, 8h in air



Annealing at 1600°C for 1min



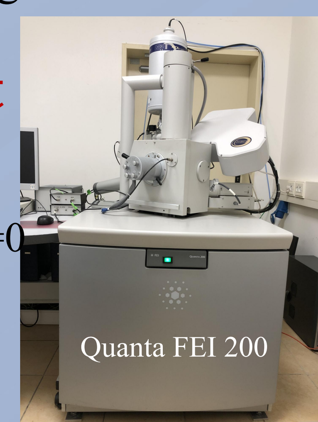
Annealing at 1600°C for 4h

Grain size measurement and GB mobility calculation

- **Grain size measurements** were conducted by using the Line Intersection Method to analyze the SEM imaging of polished and thermally etched cross sections via MATLAB[6].
- **Mobility measurements** were carried out by measuring the change in average grain size.

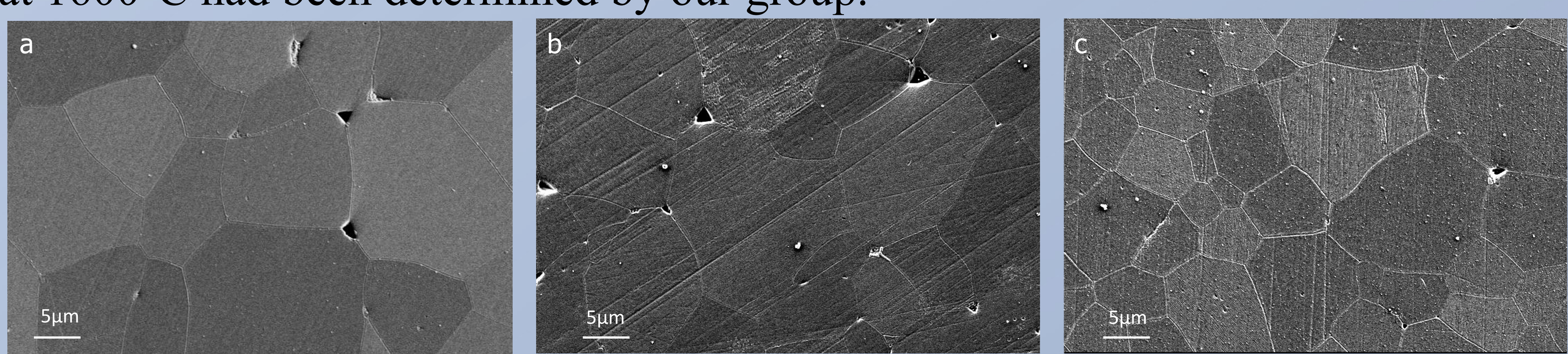
$$\bar{G}_t^2 - \bar{G}_0^2 = 4M_{GB}\gamma_{GB}t = k \cdot t$$

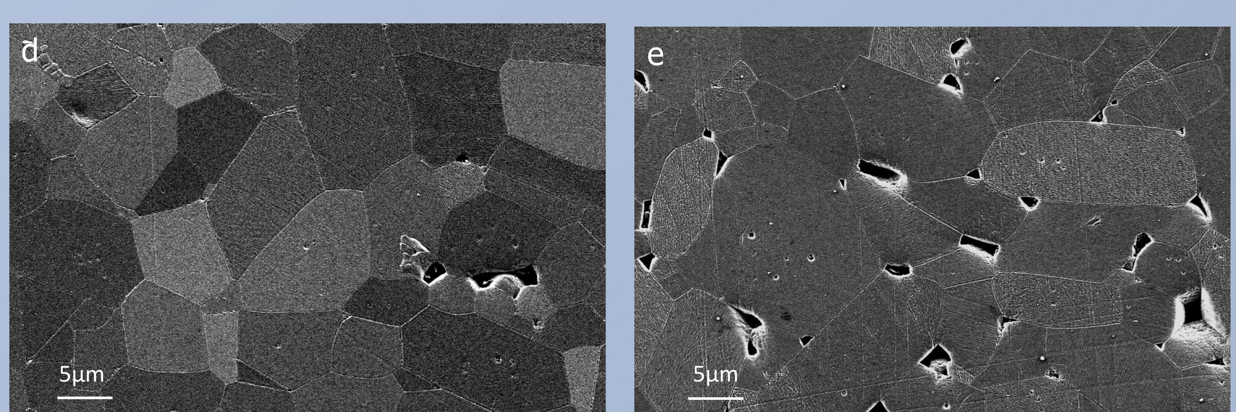
$\bar{G}_t^2$ - average squared grain size at time t
$\bar{G}_0^2$ - average squared grain size at time t=0
$M_{GB}\gamma_{GB}$ - effective GB mobility.
k - rate constant.



Results and Discussions

Solubility limit: In order to correlate the gain growth kinetics in alumina with the possible complexion tansitions rather than with precipitation of a second phase, it is critical to determine the grain boundary mobility experimentally with confirmed limit of coexistence. The solubility limit of several dopants of interest in alumina at 1600°C had been determined by our group.

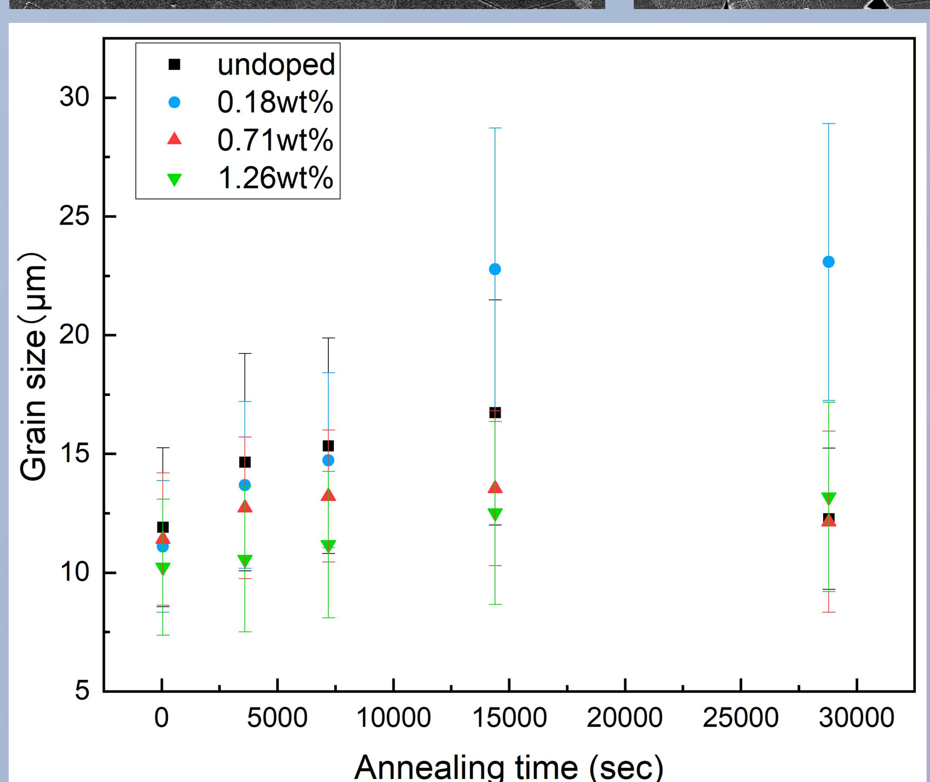


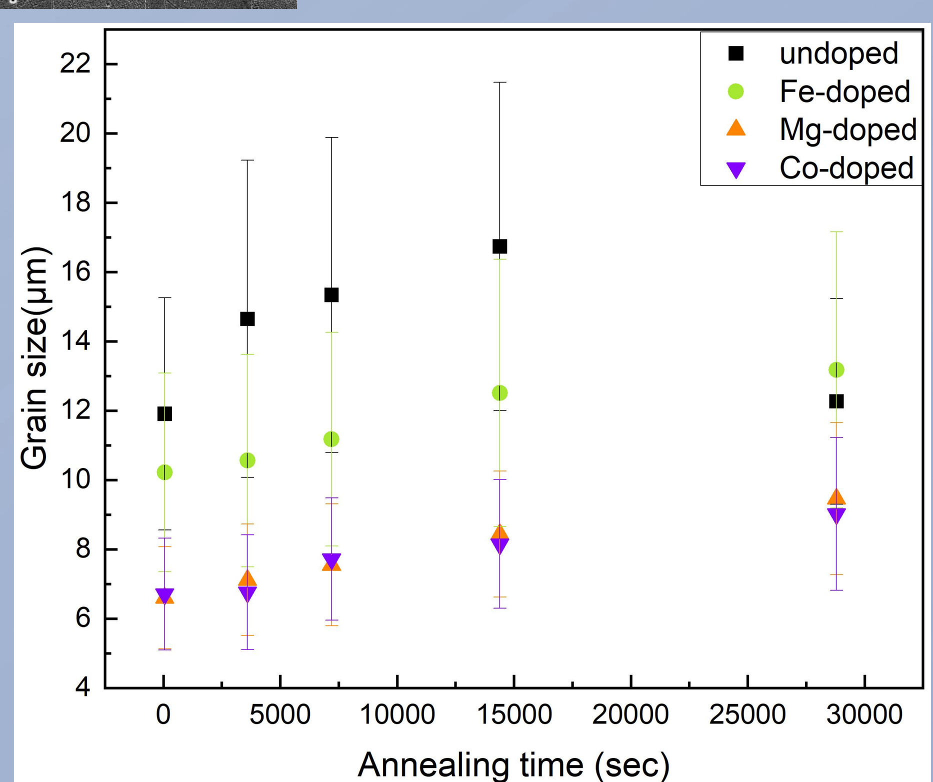


SEM micrographs (polished cross-section) of the microstructure of **undoped alumina(a)**, **0.18wt%**Fe-doped alumina **(b)**, **0.71wt%**Fe-doped alumina **(c)**, **1.26wt%**Fe-doped alumina **(d)**, and **2.93wt%**Fe-doped alumina **(e)** annealed for 2 h at 1600°C in air.

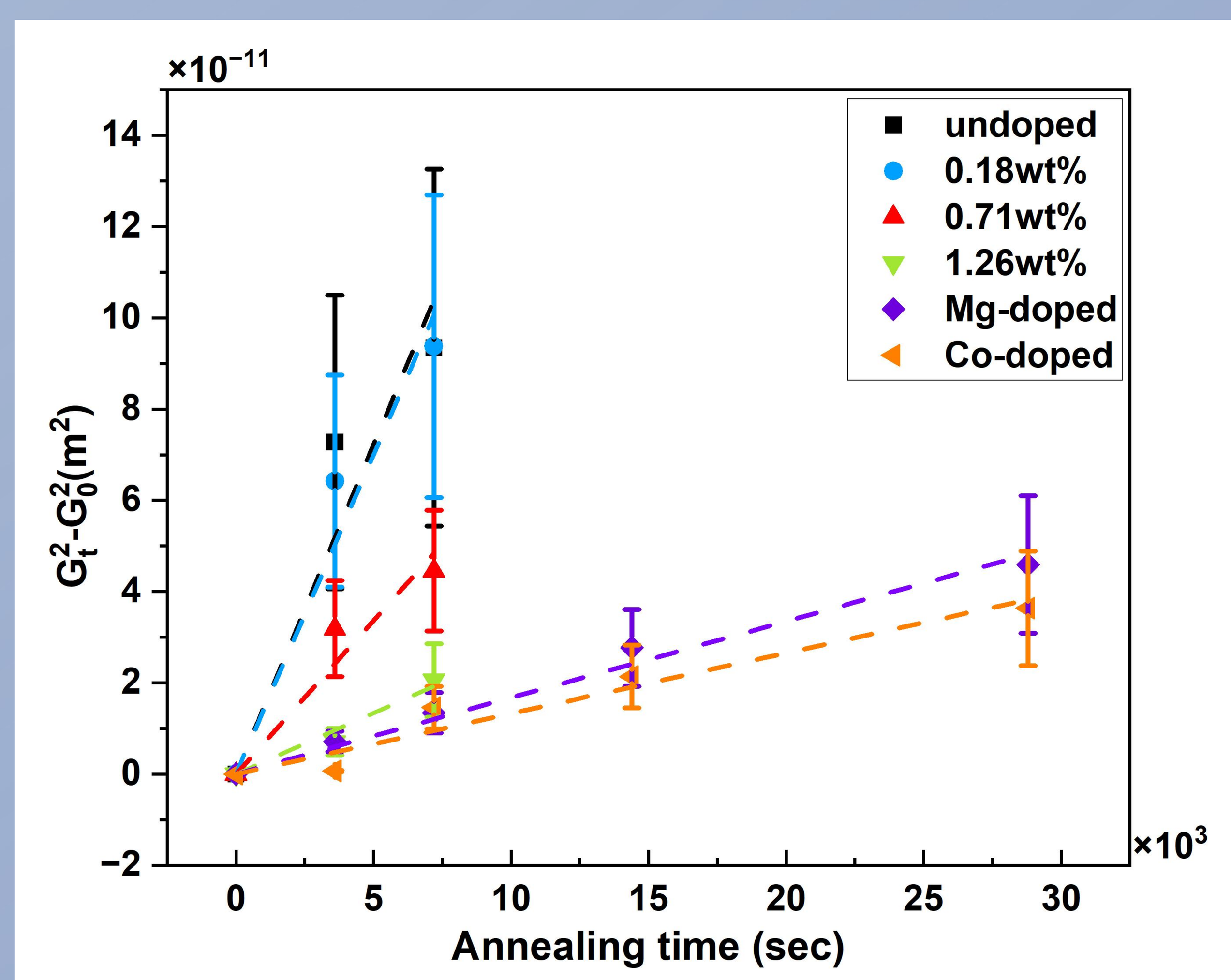
Dopant species	Solubility limit [ppm]	Effect on GB mobility(m^2/s) [below solubility limit]
Ca^{2+}[5]	51	increase
Mg^{2+}[5]	132	decrease
Cr^{3+}	no known limit	decrease
Fe^{3+}	81000[in air]	??

Grain growth





Effective GB mobility



Samples	Effective Mobility [m^2/sec]
undoped	2.80404E-15
0.18wt%	2.60578E-15
0.71wt%	1.27042E-15
1.26wt%	4.06094E-16
Mg-doped	3.24839E-16
Co-doped	2.49400E-16

Conclusions

- Doping alumina with Fe (below the solubility limit) can retard grain growth during annealing treatment.
- The alumina effective GB mobilities decrease with the increasing of Fe concentration.
- Both Mg and Fe dopants can decrease GB mobility. Mg dopants have a stronger drag effect on alumina than that of Fe dopants.
- Fe dopants under solubility limit reduce GB mobility by solute drag, similar to doping Mg and Cr.

References

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