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Microstructure evolution during high-pressure spark plasma sintering (HPSPS) of transparent alumina

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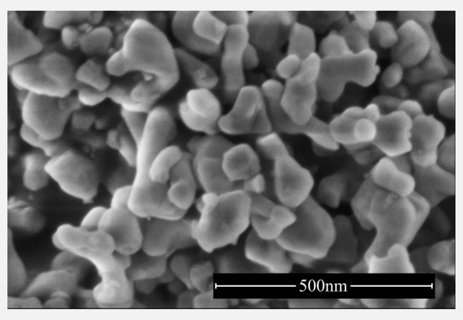
What is HPSPS?

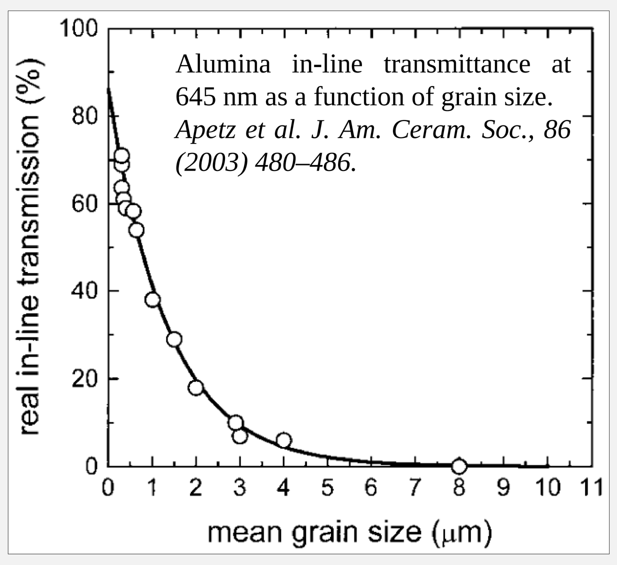
High-pressure spark plasma sintering (HPSPS) is a technique in which some modification to the standard SPS apparatus is done in a way that upgraded tooling (commonly from WC and SiC) allows to apply considerably higher pressure (usually 200-1000 MPa) during the sintering process. The higher applied pressure makes it possible to enhance the sintering process, lower the sintering temperature and end up with a final product with improved properties. HPSPS is a very effective method to fabricate high-quality polycrystalline transparent ceramics with a high level of in-line transmittance. Furthermore, the typical relatively low sintering temperatures makes it possible to obtain fine-grained microstructures and even dense nano-structured ceramics.

Fabrication of transparent alumina by HPSPS

Due to the trigonal lattice structure of α-Al_2O_3 it is susceptible to birefringence. In order to be transparent, not only must the residual porosity be minimized, but also the grain size must be kept at a fine submicron size (see graph →).

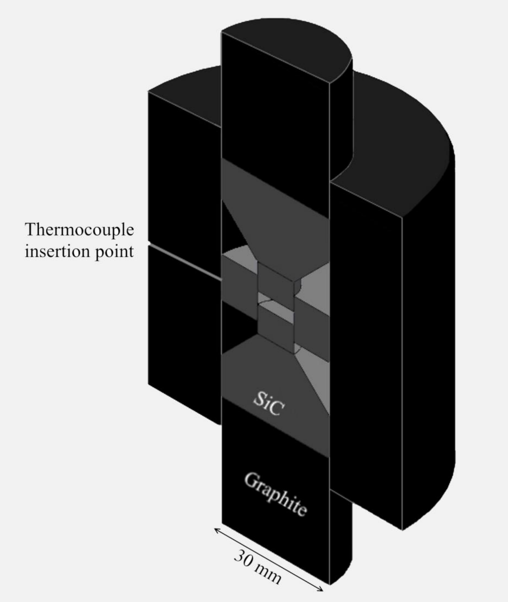


Baikowski commercial undoped and untreated alumina nano-powder (Baikalox BMA15) was used in this study.



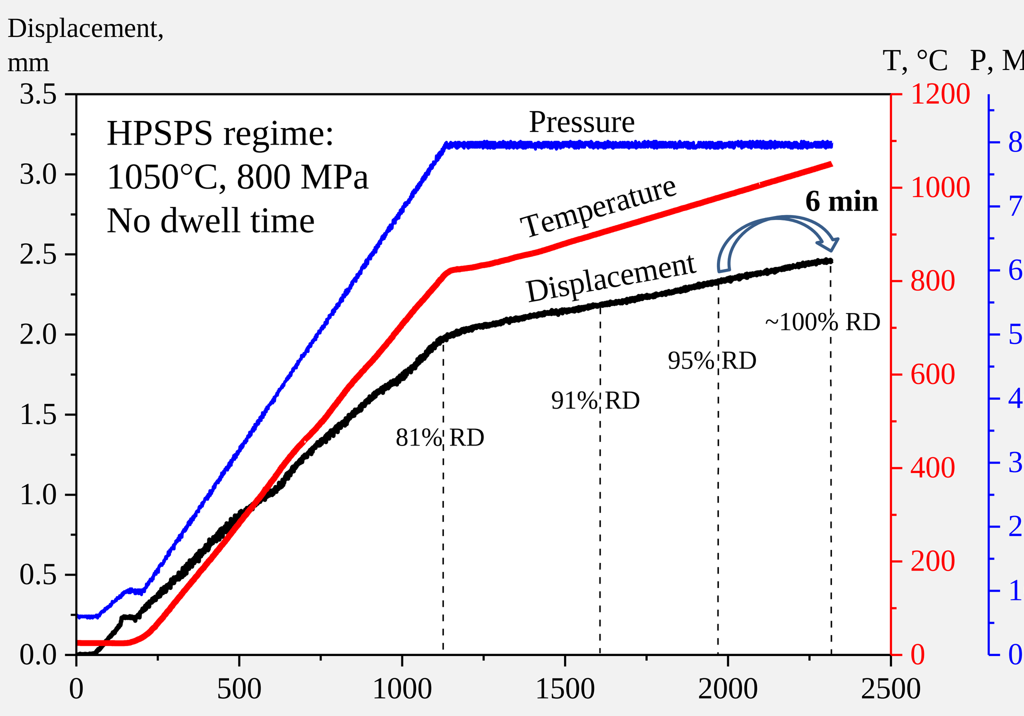
- HPSPS was performed at temperatures of 1000-1100°C, under applied pressures of 250-800 MPa and with dwell times of 0-15 min.
- Heating rates were 50°C/min up to 800°C and 12.5°C up to the designated temperature.
- Optical transmittance was measured using a UV-VIS Jasco 530 spectrophotometer.
- Microstructure was characterized by SEM, STEM and TEM

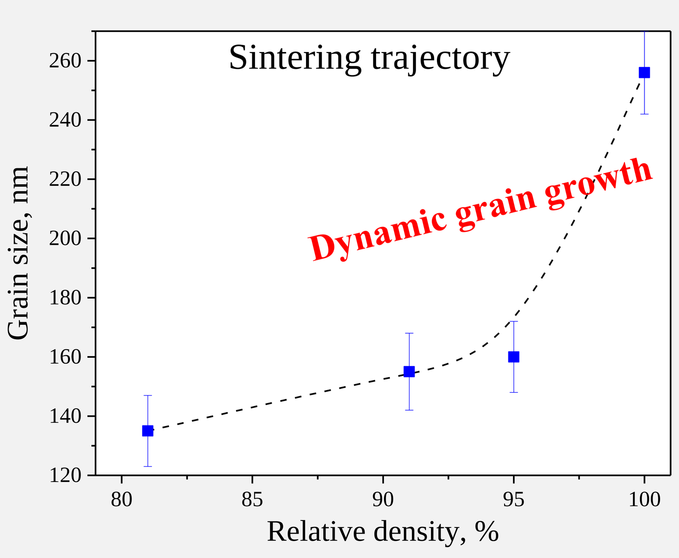
HPSPS hybrid tooling

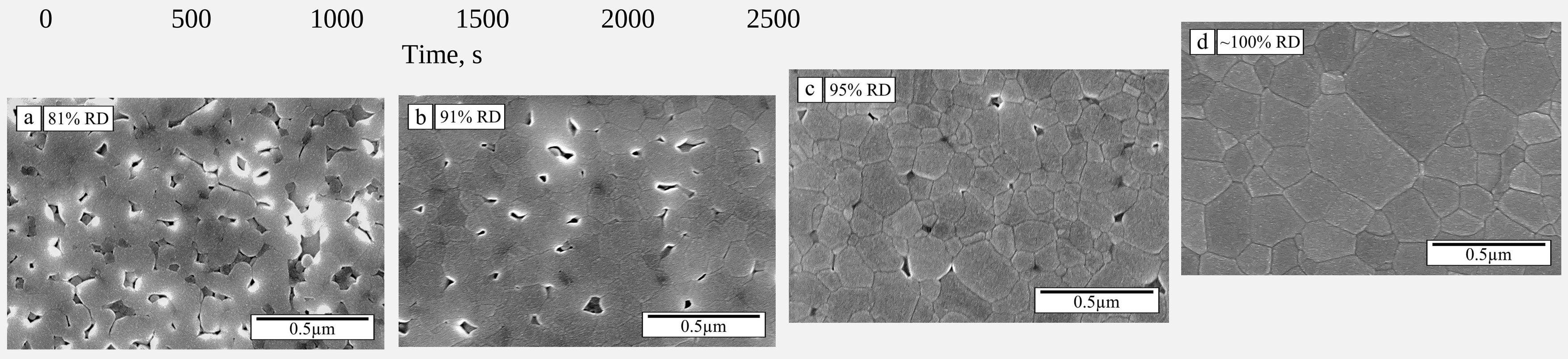


Stress-enhanced dynamic growth during HPSPS

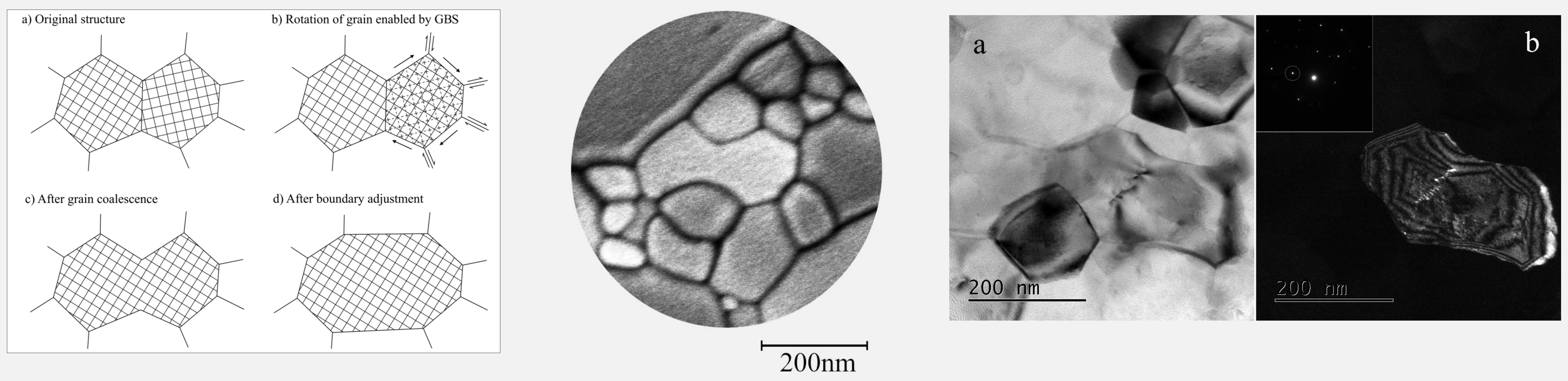
Ratzker et al., *Acta Mater*. 164 (2019) 390-399.



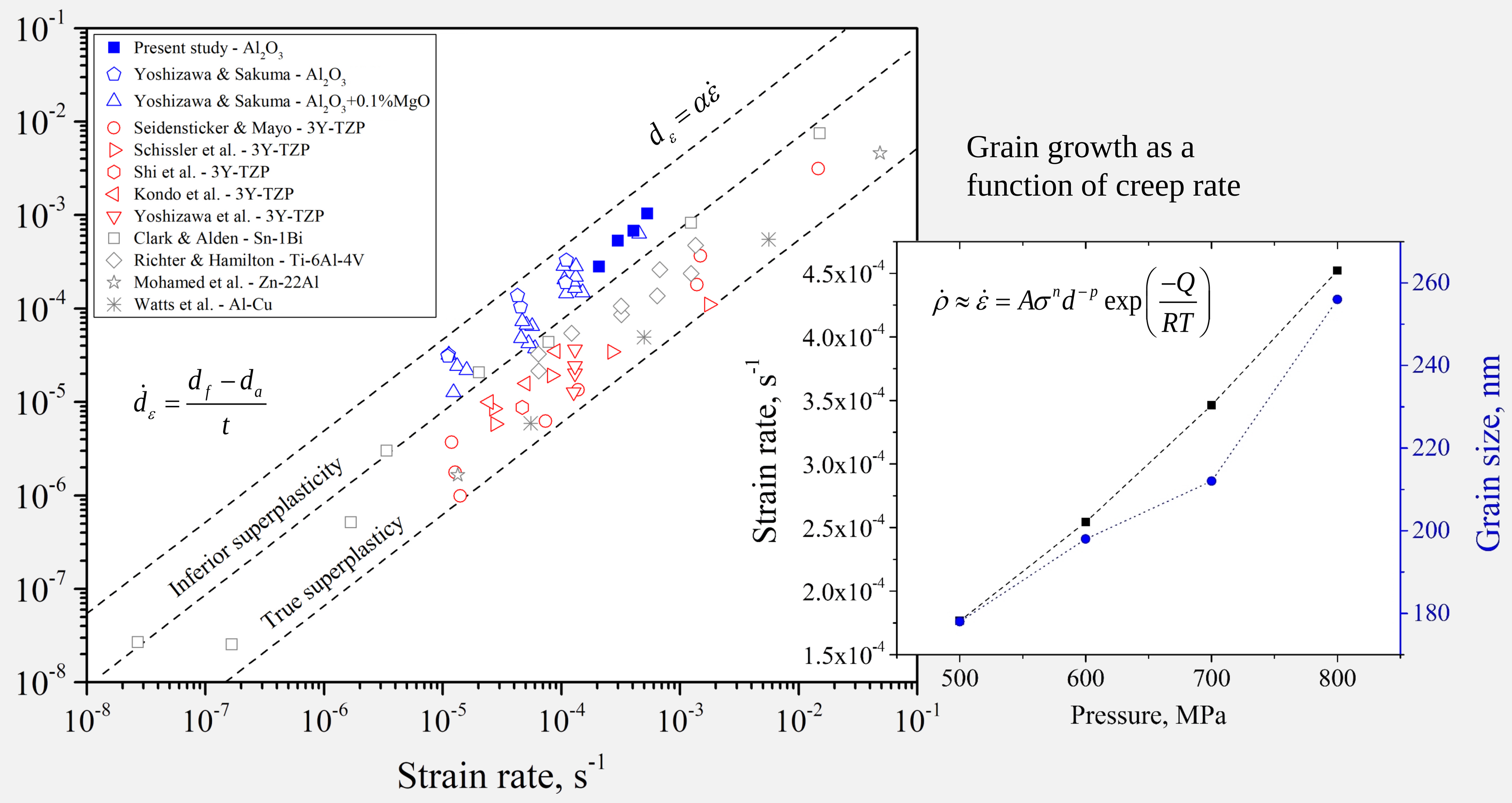




Possible grain coalescence during sintering by sliding and rotation

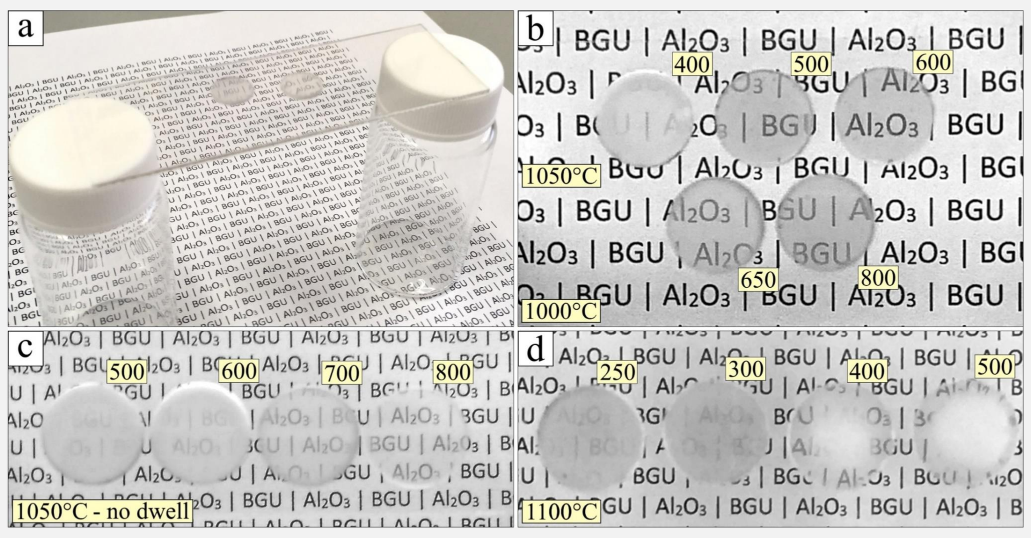


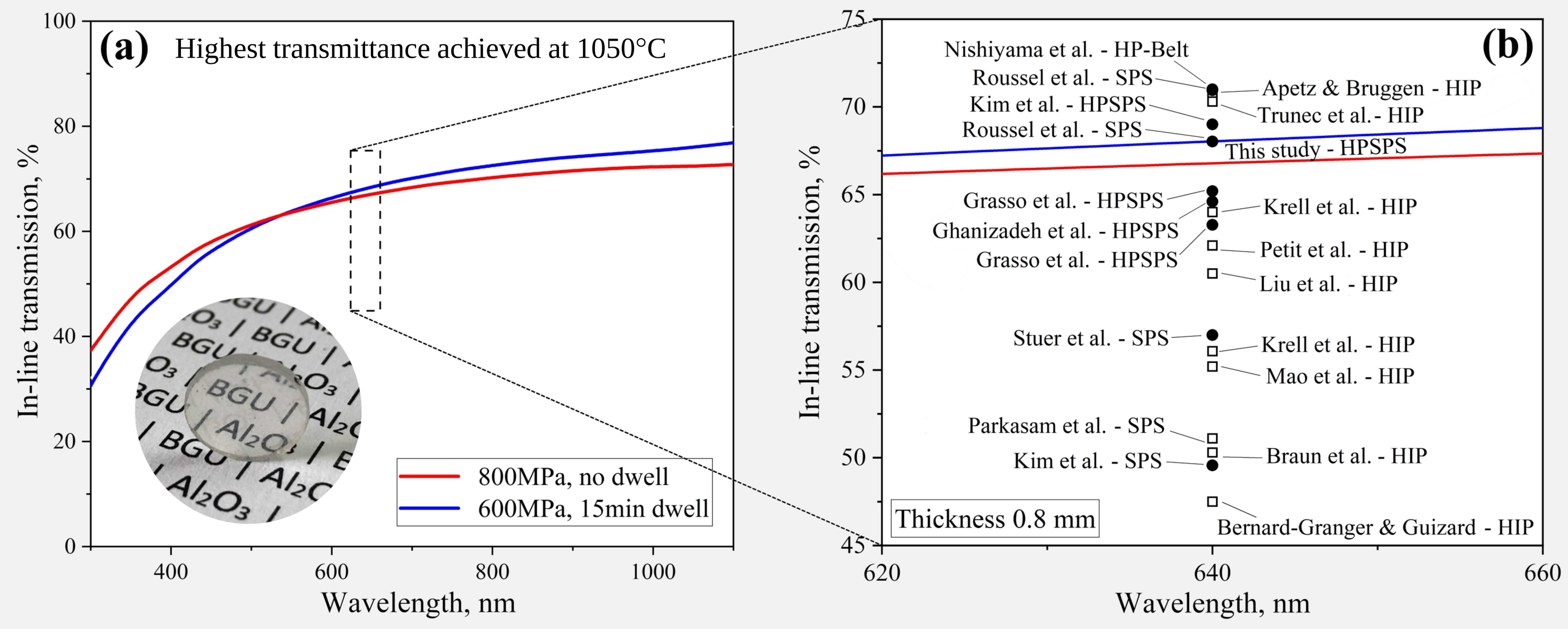
Proposed strain rate analysis resulting in stress-enhanced dynamic grain growth as in high temperature deformation of fine-grained materials



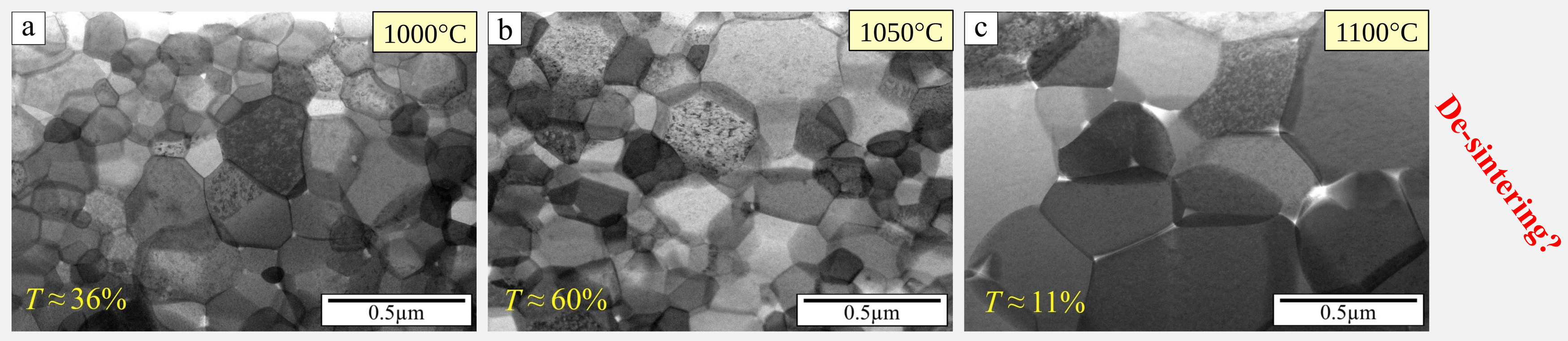
HPSPS effect on optical properties and "de-sintering"

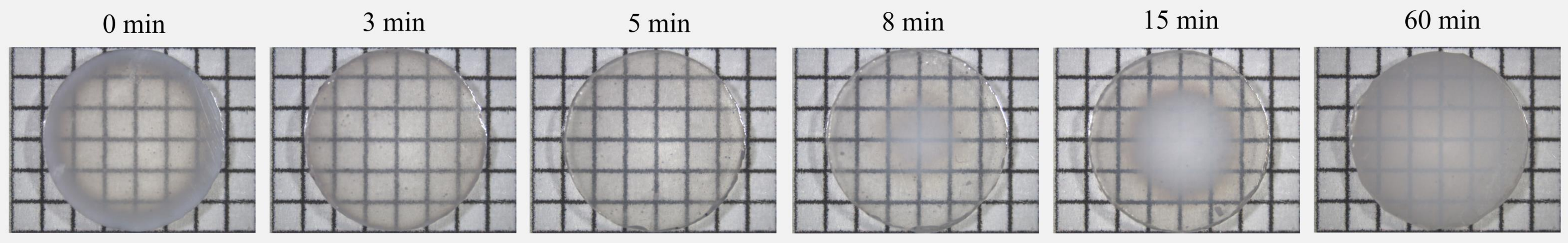
- Final grain sizes of highly transparent samples was in the range of 180-250 nm.
- Transparency and homogeneity were highly sensitive to the combination of temperature, pressure and holding time. Under certain temperature and pressure conditions, some of the samples exhibited an opaque periphery or center.
- An in-line transmittance of about 67-68% at a wavelength of 640 nm was achieved, highest results reported for SPS of **untreated commercial powder**.





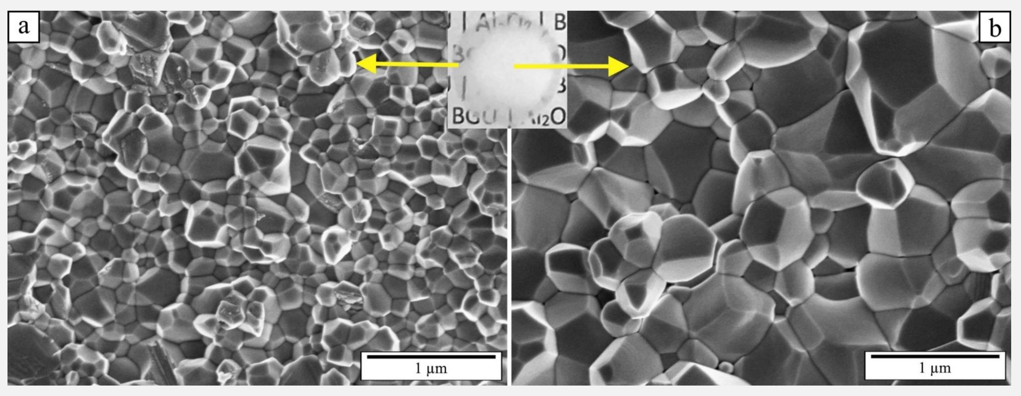
Microstructure after HPSPS at 1000-1100°C under 500 MPa with 15 min dwell time



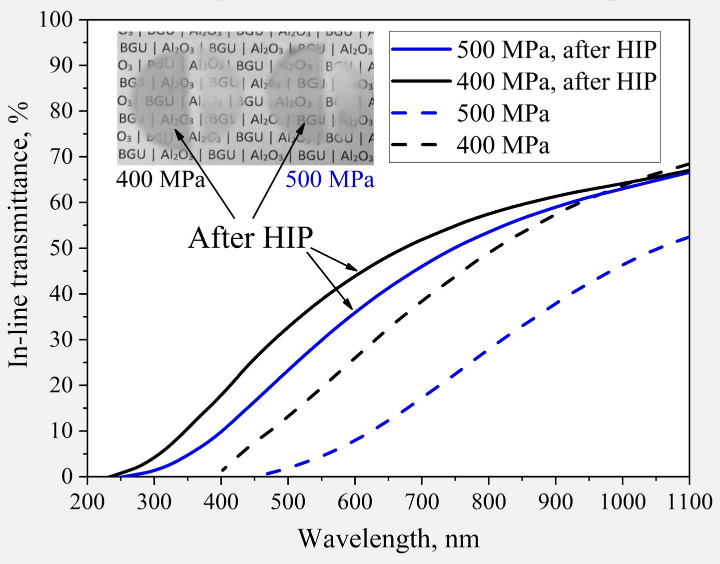


Sample appearance after HPSPS at 1100°C under 500 MPa with varying dwell time, revealing the kinetics of the process. Dwell time ⟶

"De-sintering" was accompanied by significant grain growth, notice the difference in grain size at the center and periphery of samples.



HIP (1000°C, 5 h) was performed to reclose pores of "de-sintered" samples.



Conclusions

Two major phenomena are observed during HPSPS of transparent alumina:

1. Stress-enhanced dynamic grain growth. Rapid grain growth took place during creep densification final stage of sintering under conditions in which thermally activated grain growth mechanisms were negligible. It is suggested that at higher strain rates, more defected regions progressively developed between grains by unaccommodated grain boundary sliding, which induced enhanced grain growth. Furthermore, grain coalescence by rotation and sliding my be a contributing factor as well.

2. "De-sintering". Creep proceeds during extended dwell times when samples are subjected to relatively excessive temperature or pressure leading to cavitation and pore growth. This phenomena strongly affects the level of in-line transmittance.