

Influence of powder characteristics on cold sintering of nano-sized ZnO with density above 99%

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1. Supplementary information related to powders' characterization

This section provides some further details of the characterization of the as received ZnO powders used during presented cold sintering studies.

Table S1 is showing the results of ICP-OES (inductively coupled plasma with optical emission spectroscopy) of the as received ZnO powders.

Table S1. ICP-OES of as received powders, ZnO_20-30 and ZnO_40-100

ICP-OES (mean concentration with standard deviation) for ZnO powders					
Elements	Zn	Mg	Na	P	Ti
ZnO_20-30	76.4 ±1.5	0.0054 ±0.0004	0.663 ± 0.015	0.526 ±0.012	0.045±0.002
ZnO_40-100	81 ± 3	<0.0001	<0.001	<0.02	<0.0002

Figure S1 is showing the X-ray diffraction (XRD) analysis with the reference of ICDD PDF 00-036-1451 and Differential thermal analysis/Thermogravimetric analysis (DTA/TGA) of the as received commercial ZnO powders.

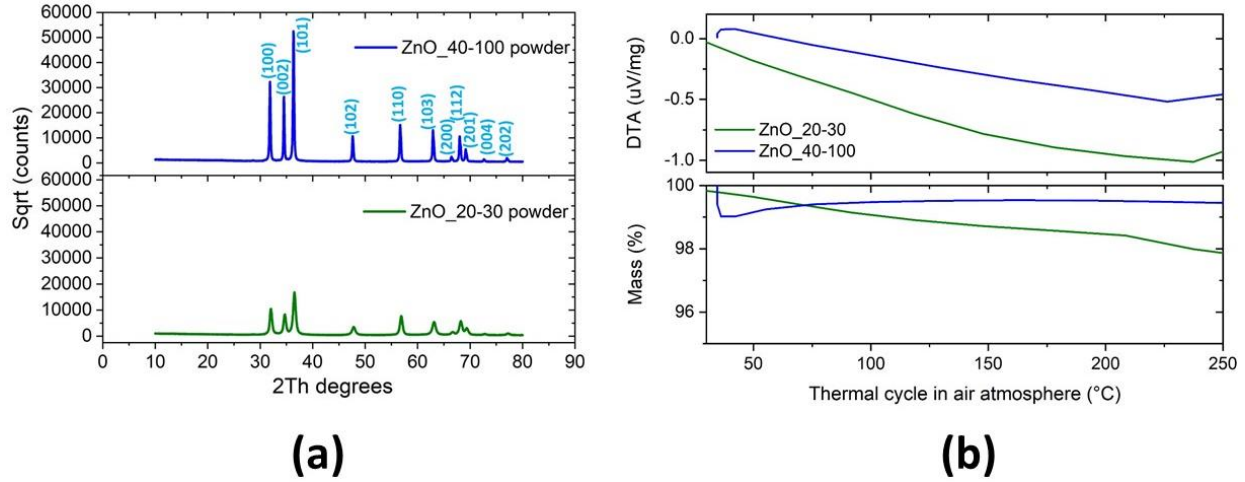


Figure S1. (a) XRD analysis of as received ZnO powder (b) DTA/TGA coupled with mass spectrometer, of as received powders (ZnO_20-30 and ZnO_40-100) thermal cycle up to 250 °C in air atmosphere with a heating rate of 20 °C/min showing different mass losses from both powders

2. Supplementary information related to results of cold sintering

This section provides some complementary details related to interpret the cold sintering behavior of ZnO powder inside FAST/SPS system.

Figure S2 a is showing a comparative influence of high (100 °C/min) and slow heating rate (20 °C/min) used for cold sintering of ZnO_40-100 (at 250 °C at 50 MPa in vacuum atmosphere) inside FAST/SPS on densification behavior and the final cold sintered density. On the other hand, Figure S2 b is showing the variation in gas pressure inside FAST/SPS chamber as effected by these different heating rates used in FAST/SPS.

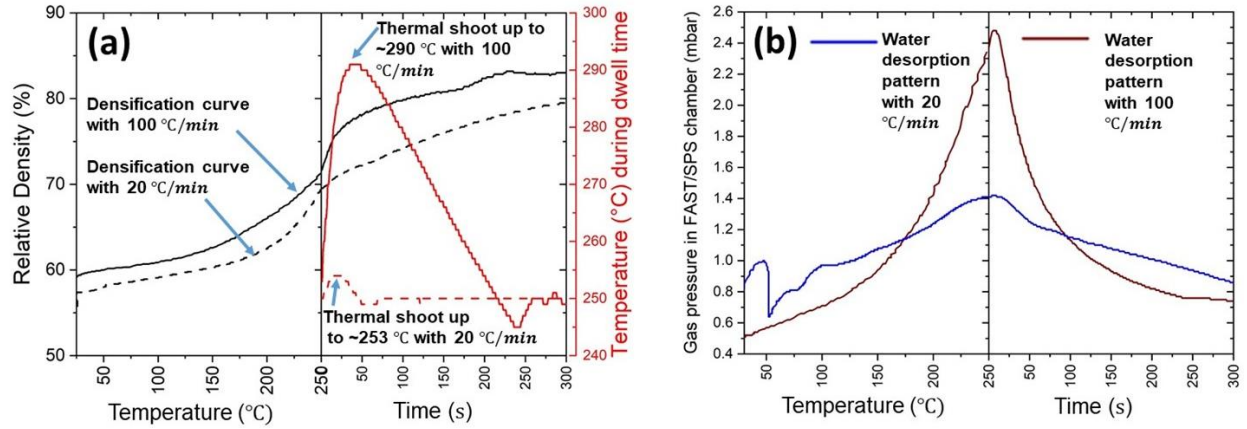


Figure S2. (a) Comparison of thermal shoot up in the dwelling time as affected by fast (100 °C/min) and slow (20 °C/min) heating rates, (b) Gas pressure variation (in FAST/SPS sintering chamber) as affected by two different heating rates of 100 °C/min and 20 °C/min

Figure S3 is showing the XRD pattern of cold sintered ZnO_40-100 pellets in three different atmospheres inside FAST/SPS with the comparison of the as received ZnO_40-100 powder. Cold sintering parameters were 250 °C, 300 MPa, 20 °C/min of heating rate in respective atmospheres using 3.2 wt. % of deionized water as an aqueous phase.

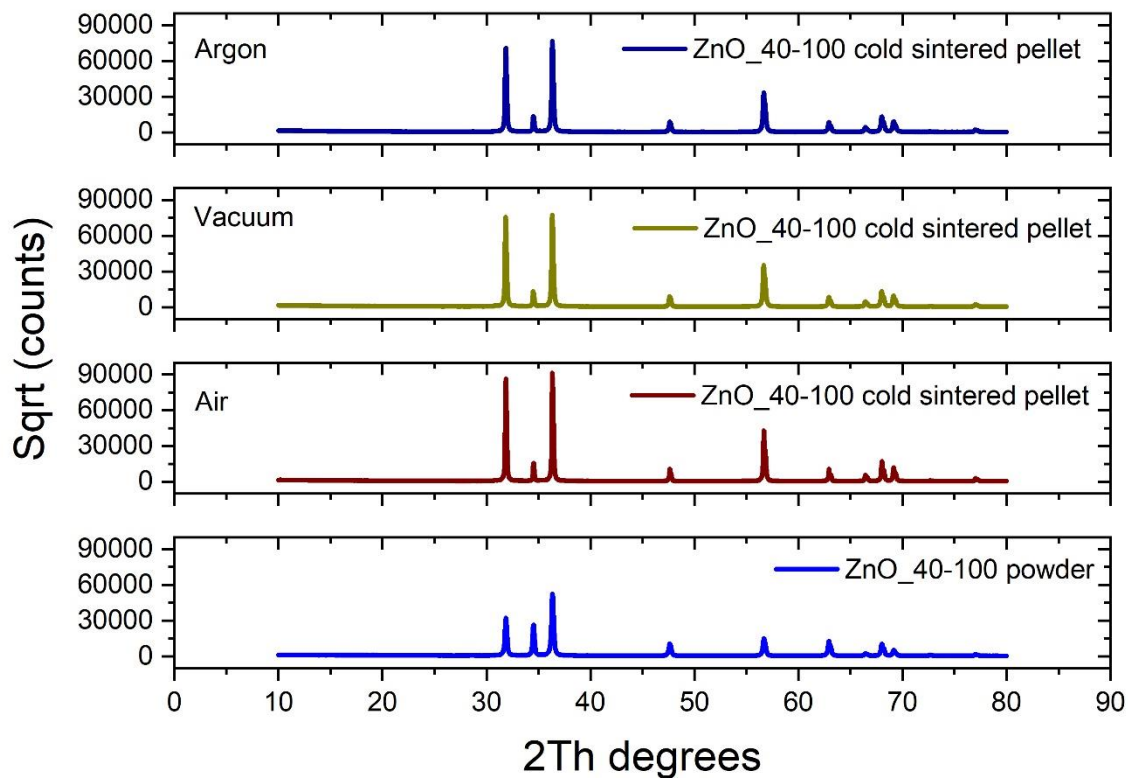


Figure S3. XRD pattern of cold sintered ZnO₄₀₋₁₀₀ in respective atmospheres (Cold sintering parameters: 250 °C, 300 MPa, 20 °C/min, 3.2 wt. % of deionized water as an aqueous phase, 10 min dwell time).

Glossary

1. Cold Sintering Process (CSP)
2. Field Assisted Sintering/Spark Plasma Sintering (FAST/SPS)
3. X-ray diffraction (XRD)
4. Brunauer–Emmett–Teller (BET)
5. Inductively coupled plasma with optical emission spectroscopy (ICP-OES)
6. Differential thermal analysis/Thermogravimetric analysis (DTA/TGA)
7. Particle/agglomerate size distribution (PSD)
8. Ultrasonic (US) agitation
9. Scanning electron microscopy (SEM)

10. Transmission electron microscopy (TEM)
11. Conventional sintering
12. Archimedes density
13. Green density
14. Wet density
15. Cold sintered density
16. Conventional sintered density
17. Coefficient of friction
18. Angle of repose
19. Settled apparent density
20. Compaction factor B
21. Image analysis
22. Lineal intercept method
23. ImageJ software
24. Thermal shoot up
25. Densification curves
26. Gas pressure variation curves