

# Microstructure of Ni-SiC Nanocomposites

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## Introduction

- ❖ Ceramic nanocomposites are ceramic-based materials reinforced with sub-micron sized particles of a second phase [1]. Nanocomposites have been reported to have improved mechanical properties compared to monolithic materials [2]. The method of forming nanometer-sized metal particles within a ceramic matrix by reducing infiltrated salts was first demonstrated on the Ni-alumina system [3].
- ❖ SiC has many polytypes. The five most common are: 6H-SiC, 4H-SiC, 2H-SiC, 3C-SiC and 15R-SiC.
- ❖ Nickel reacts with SiC to form silicides at the interface [4]. Various silicides form according to the Ni-Si phase diagram as shown in Figure 1 [5]. The main silicides are: Ni<sub>2</sub>Si, Ni<sub>3</sub>Si, NiSi and NiSi<sub>2</sub> [4] as described in Table 1.

## Experimental Methods

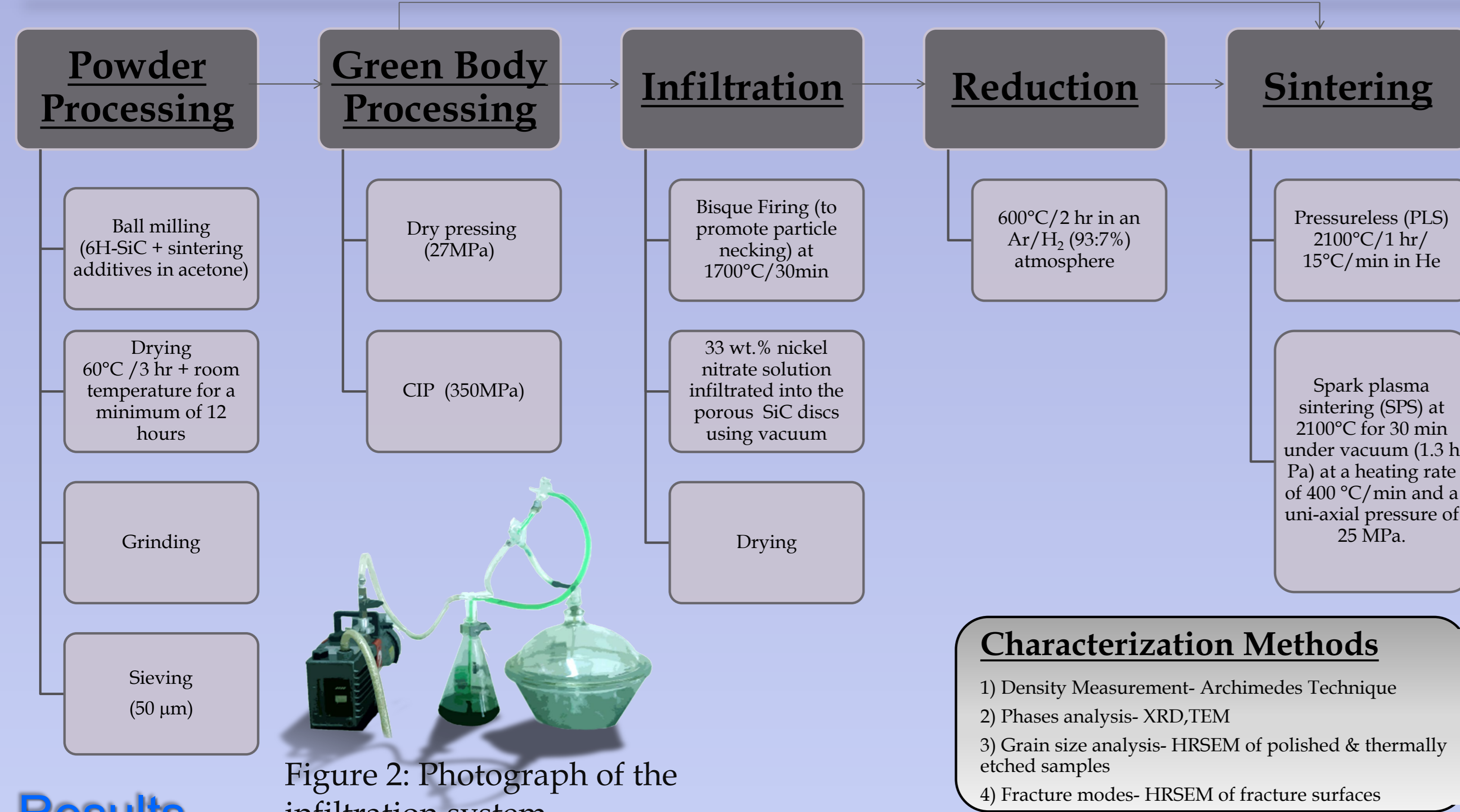


Figure 2: Photograph of the infiltration system.

## Results

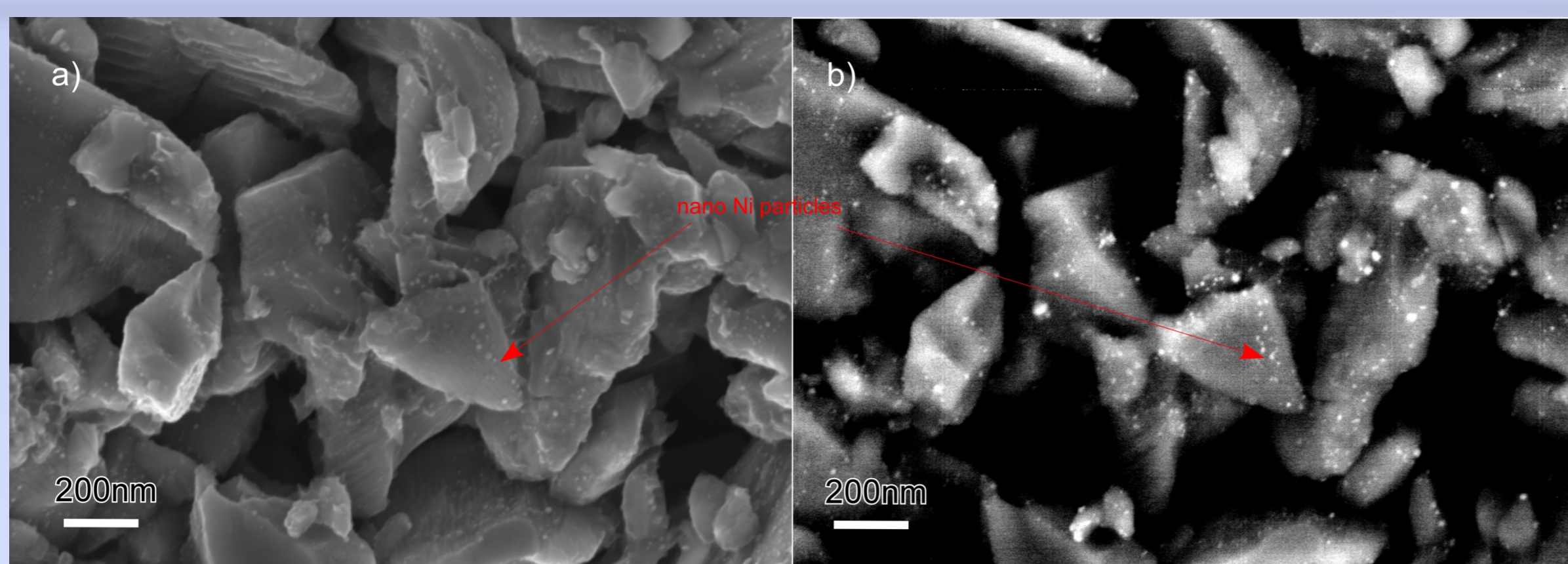


Figure 3: HRSEM micrographs of a bisque-fired sample after infiltration and reduction (prior to sintering). a) Secondary electrons (SE) and b) backscattered electrons (BSE).

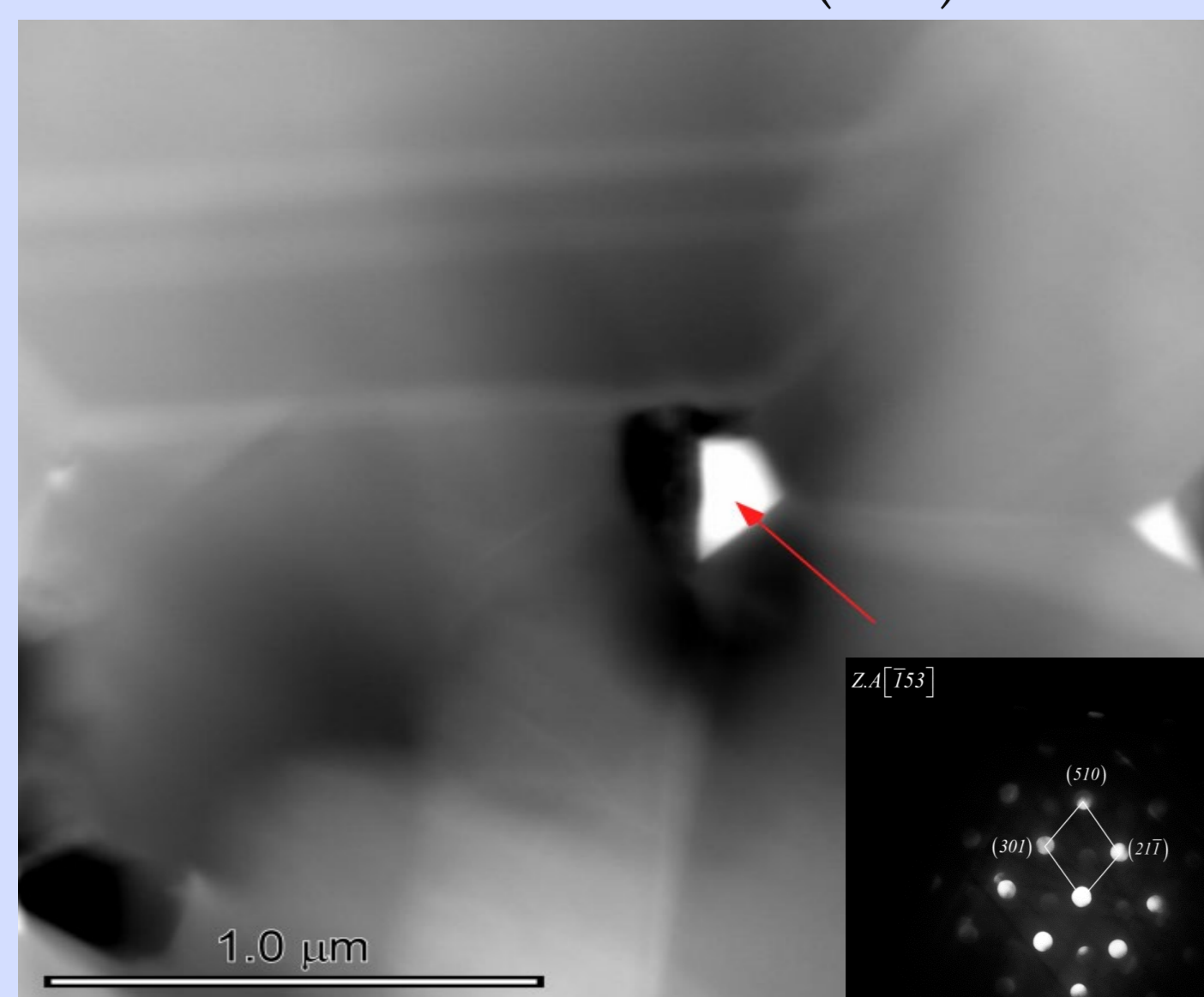


Figure 4: Dark field STEM micrograph of a PLS sample showing a silicide particle at a triple junction, and a CBED pattern of the particle (Ni<sub>2</sub>Si) in a [153] zone axis.

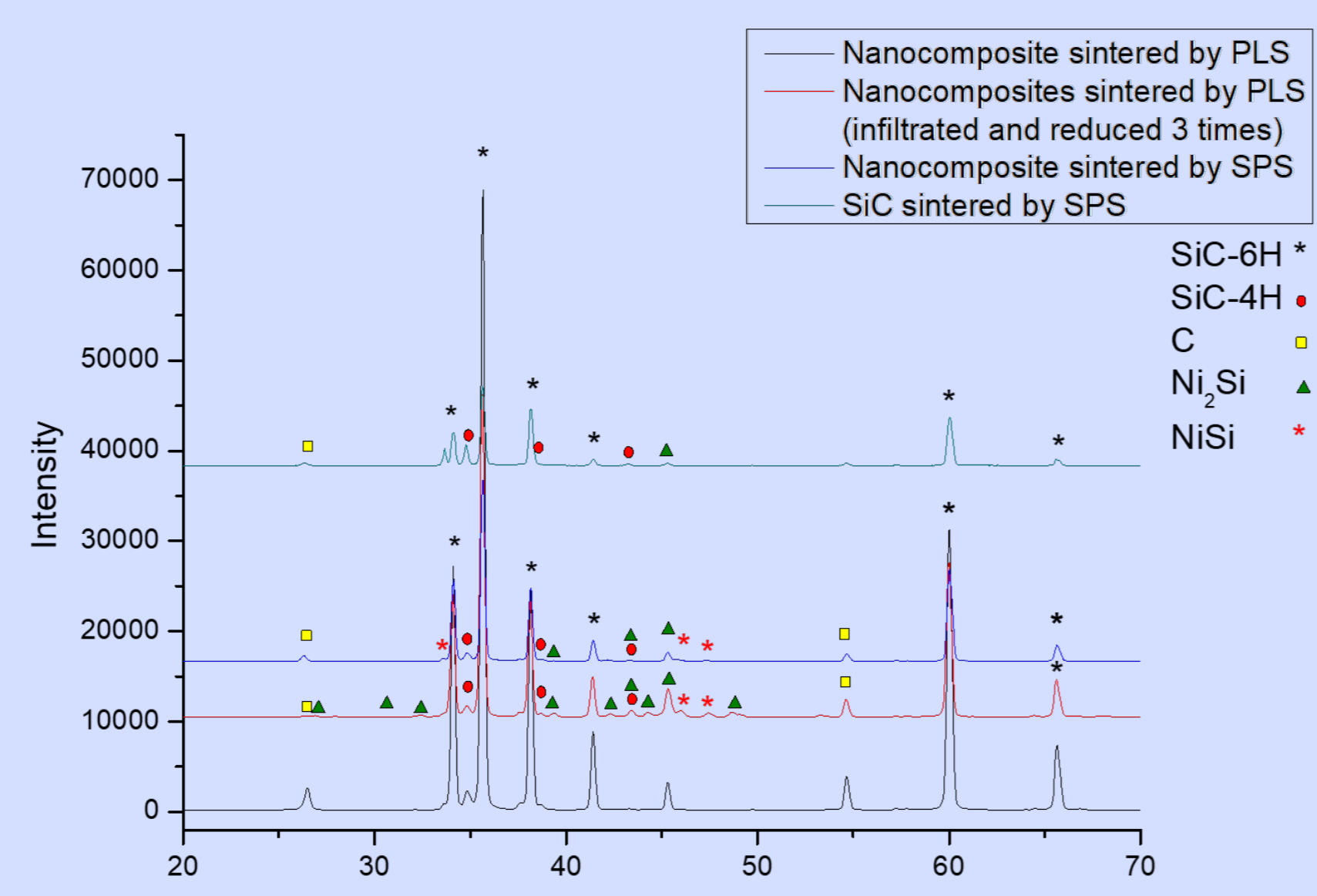


Figure 5: XRD of the different samples indicating the presence of 6H-SiC, 4H-SiC and C phases in all samples, as well as Ni<sub>2</sub>Si and NiSi in the nanocomposites.

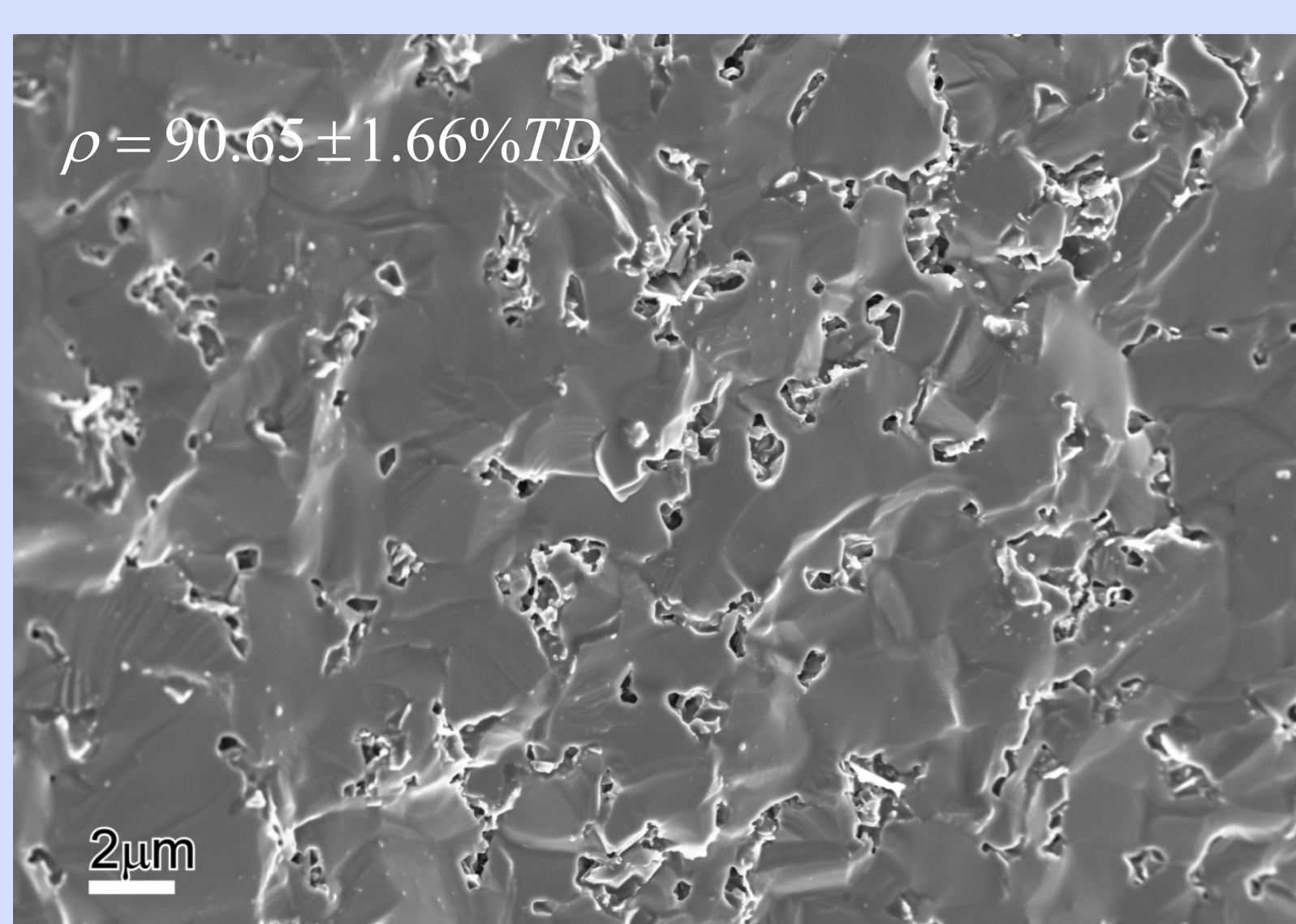


Figure 6: SE SEM micrograph of a nanocomposite sintered by PLS.

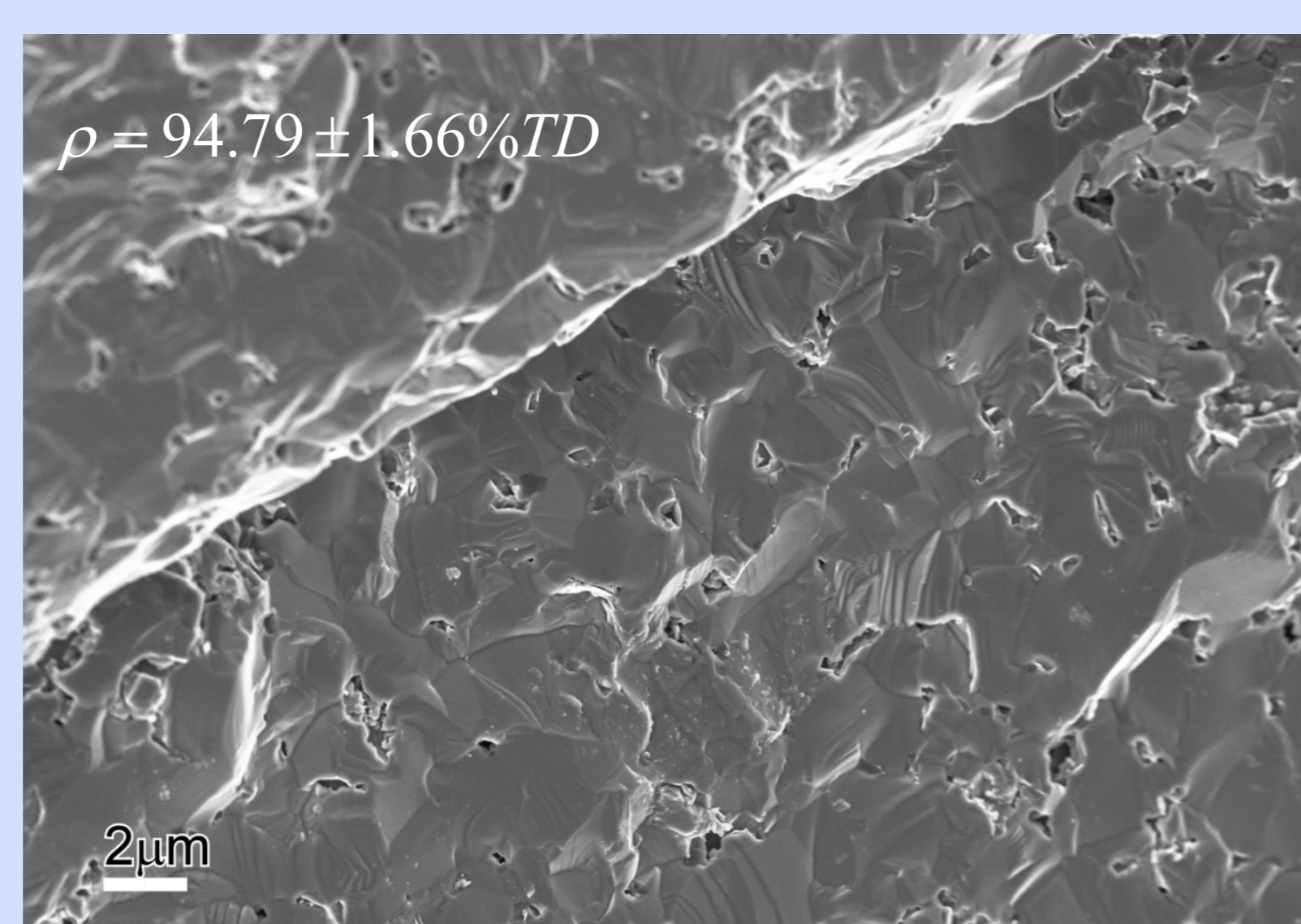


Figure 7: SE SEM micrograph of pure SiC (control sample) sintered by PLS.

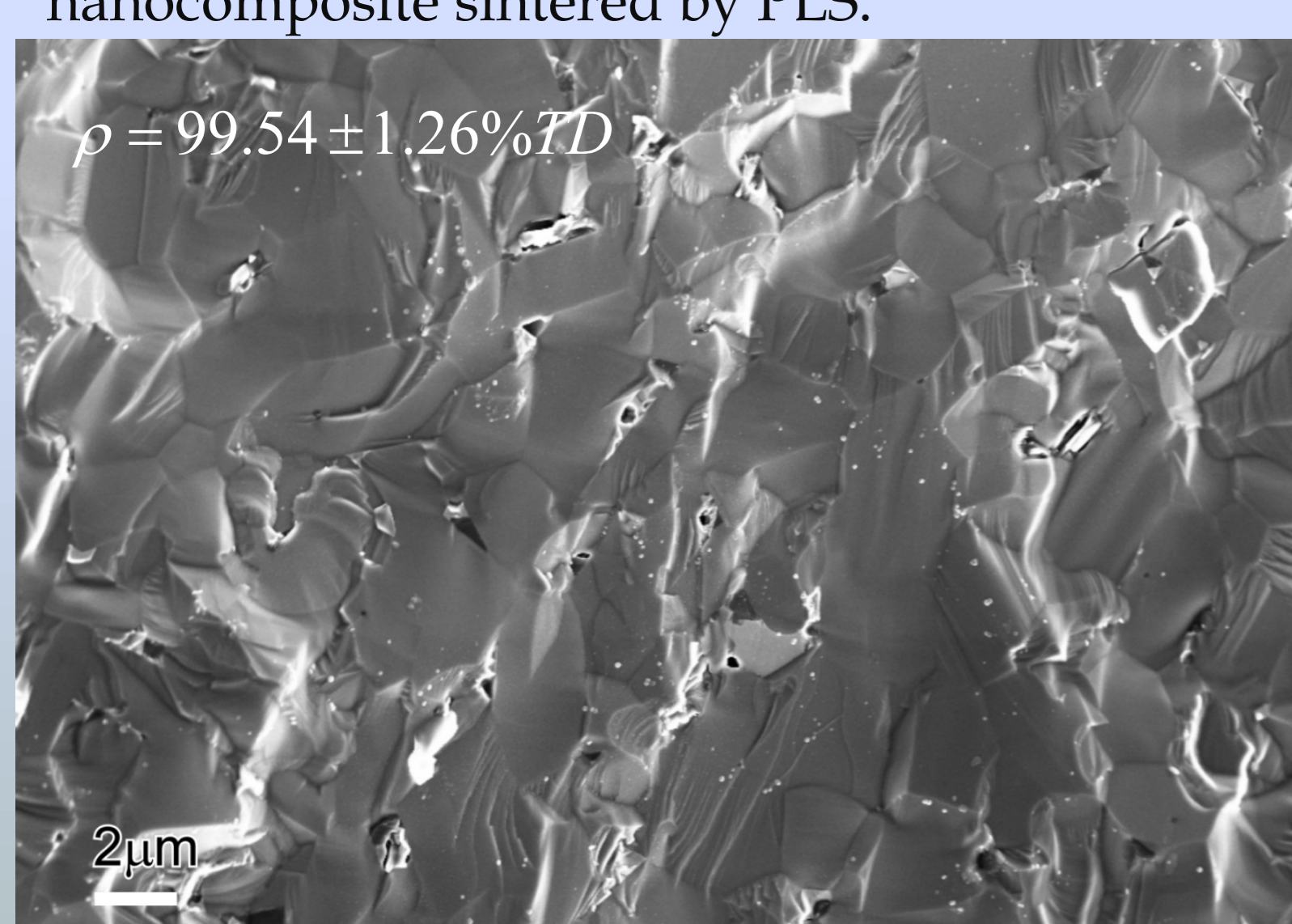


Figure 8: SE SEM micrograph of nanocomposite sintered by SPS.

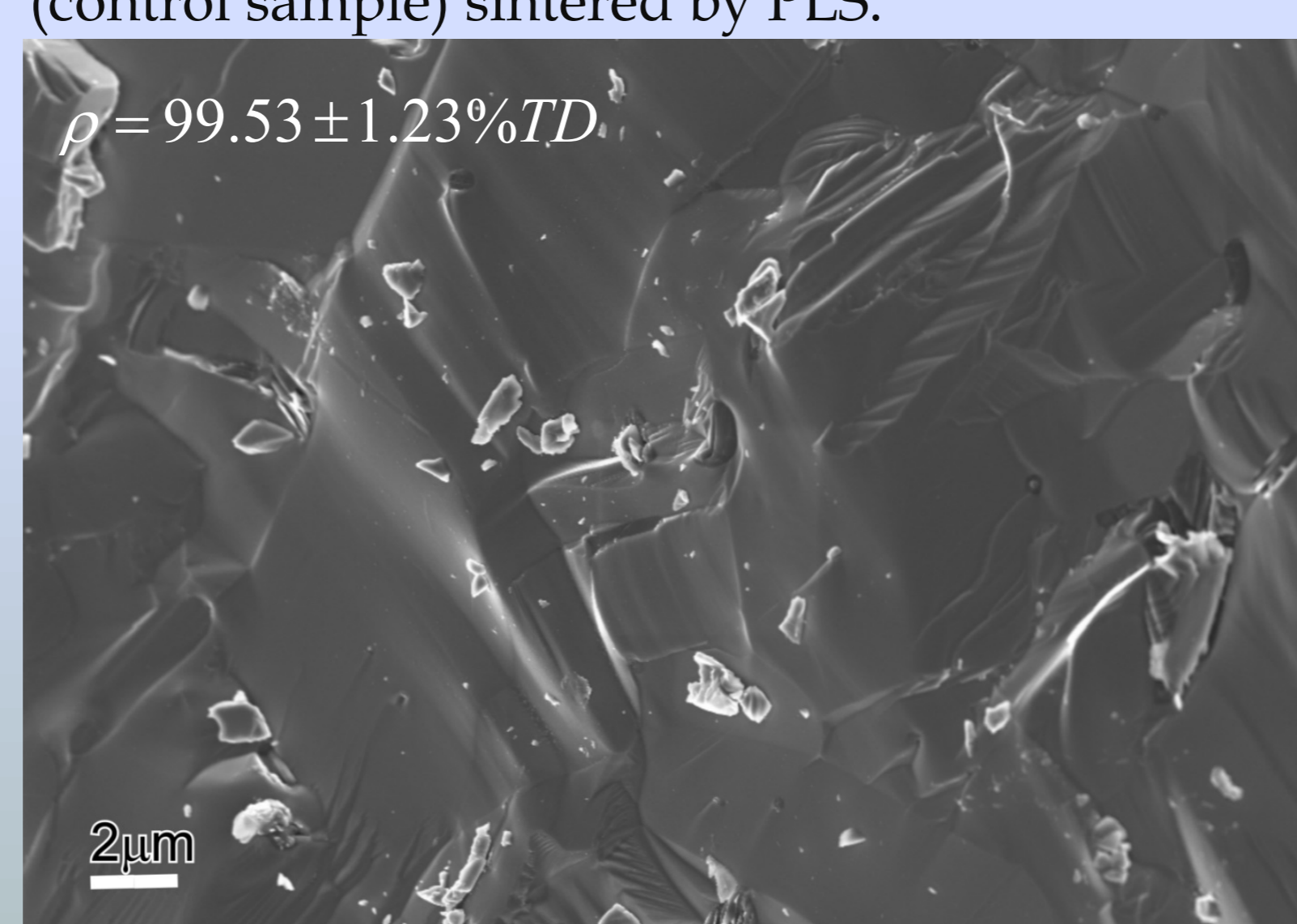


Figure 9: SE SEM micrograph of pure SiC (control sample) sintered by SPS.

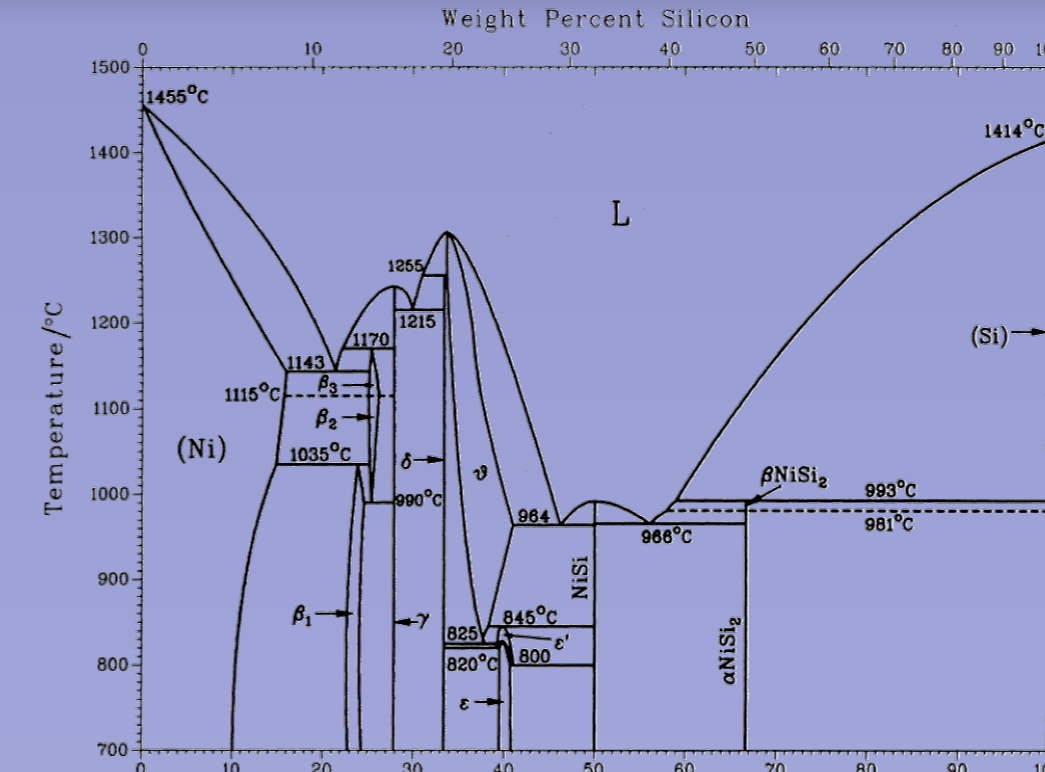


Figure 1: Ni-Si phase diagram[5].

Table 1: Possible Ni-SiC reactions and their Gibbs' energies [4].

Reaction	Gibbs' energy as function of temperature (KJ/mol Ni)
$Ni + 2SiC \rightarrow NiSi_2 + 2C$	$22.990 + 0.0108T \log T - 0.0454T$
$Ni + SiC \rightarrow NiSi + C$	$-30.932 + 0.0054T \log T - 0.0195T$
$Ni + 2/3SiC \rightarrow 1/3Ni_3Si + 2/3C$	$-38.317 + 0.0036T \log T - 0.0158T$
$Ni + 1/2SiC \rightarrow 1/2Ni_2Si + 1/2C$	$-41.800 + 0.0027T \log T - 0.0119T$

## Research Goals

- ❖ To produce SiC reinforced with Ni-based particles, produced by pressureless sintering (PLS) or spark plasma sintering (SPS).
- ❖ To evaluate the differences in the microstructure of the Ni-SiC nanocomposites as a function of the applied sintering method.

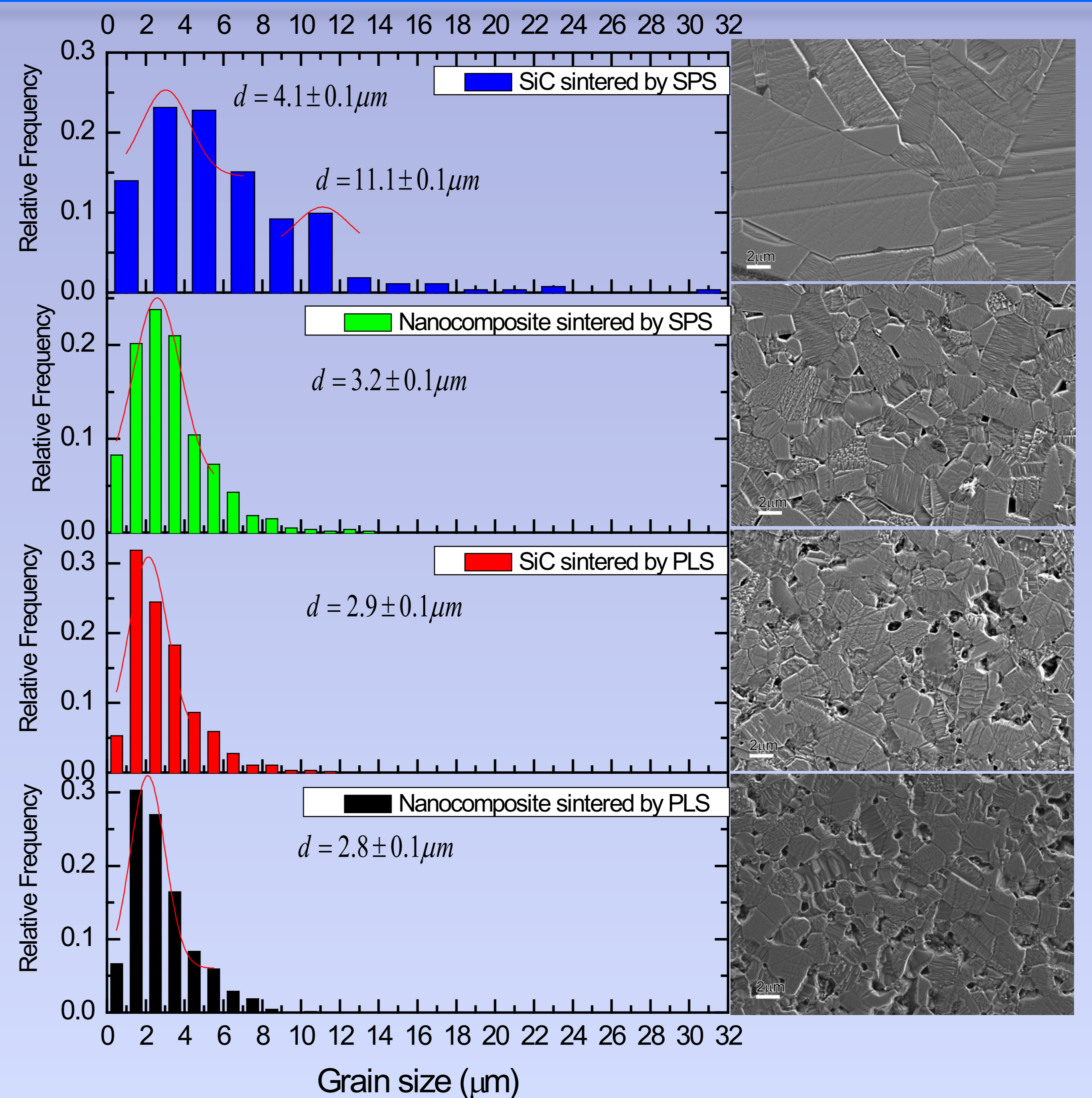


Figure 10: Histogram analysis of the SiC grain size for the different samples. 600 grains were measured for each sample, except for pure SiC (control sample) sintered by SPS (300 grains).

- ❖ The dominant silicide phase in the nanocomposites is Ni<sub>2</sub>Si, which has a larger negative Gibbs' energy at temperatures above 1500°C (see Table 1). Traces of NiSi, which has a less negative Gibbs' energy, indicate that a possible cored microstructure exists for the particles. Silicide formation is envisioned to commence with Si and C diffusing into the Ni due decomposition of SiC. The silicide starts to form at the interface. Due to the lack of Si in the center of the Ni particles, NiSi forms instead of Ni<sub>2</sub>Si. As the temperature raises more Si diffuses towards the center of the particles, and the NiSi phase transforms into Ni<sub>2</sub>Si, which is more thermodynamically stable. The particles are larger after SPS compared to PLS.
- ❖ According to fracture surface analysis, more transgranular fracture is observed in the PLS nanocomposite compared to the control sample (SiC sintered by PLS). There is less transgranular fracture in the SPS nanocomposite compared to its control sample. Furthermore, more transgranular fracture is observed in the SPS nanocomposites and control samples compared to the nanocomposites and control samples prepared by PLS.
- ❖ The microstructure of the nanocomposites sintered by PLS or by SPS, and that of the SiC control sample sintered by PLS are uniform with an average grain size of ~3µm. Abnormal grain growth can be observed in the SiC control samples sintered by SPS. This indicates that the silicide phase in the nanocomposite sintered by SPS prevents abnormal grain growth during sintering. Furthermore, it can be seen that the silicide particles in the nanocomposite processed by SPS are dispersed homogeneously.

## Conclusions

- ❖ The dominant silicide phase in the nanocomposite is Ni<sub>2</sub>Si.
- ❖ The silicide particles have a cored microstructure, with the main silicide being Ni<sub>2</sub>Si at the outer rim (in contact with SiC), and NiSi in the core.
- ❖ The silicide particles prevent abnormal grain growth and contributes to the homogenous microstructure of the nanocomposite sintered by SPS. Abnormal grain growth occurs in the SiC control samples, sintered by SPS, resulting in a larger average grain size and a less homogeneous microstructure.

## Acknowledgments

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## References

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