

BACKGROUND

NiS₂

- Crystallizes in pyrite structure with +4 oxidation state
- Sulfur removal through HDS reactions for oil refining
- MoS₂ promoter in HDS reactions
- Other techniques used to synthesize nanoparticles

Microwave Irradiation

- Applied to compounds other than NiS₂
- Increased reaction rate
- Small deviations in grain size

PURPOSE AND HYPOTHESIS

- Green chemistry
- Low temperatures and rapid rate

This experiment performs the hydrothermal synthesis of NiS₂ nanoparticles which will be analyzed via XRD and SEM to determine average particle size, composition, and morphology.

MATERIALS AND METHODS

Approximately a 1:1 ratio of nickel nitrate hexahydrate and sodium thiosulfate were dissolved in 500mL on 18MΩ deionized water. The mixture was divided into equal portions and placed in a Teflon lined digestion bomb. Using microwave irradiation, the solution was heated to the desired temperature, cooled, filtered, and washed with portions of 18MΩ deionized water, methanol, and acetone. The solution ran at 6 different temperatures from 120°C to 200°C at 20° increments for 30 minutes each. In addition, the solution ran at 180°C for 2 hours.

RESULTS

- Intensity increase
- 2 phases present
- Ni₉S₈
- Stabilization at higher temperatures
- Increase in particle size with temperature

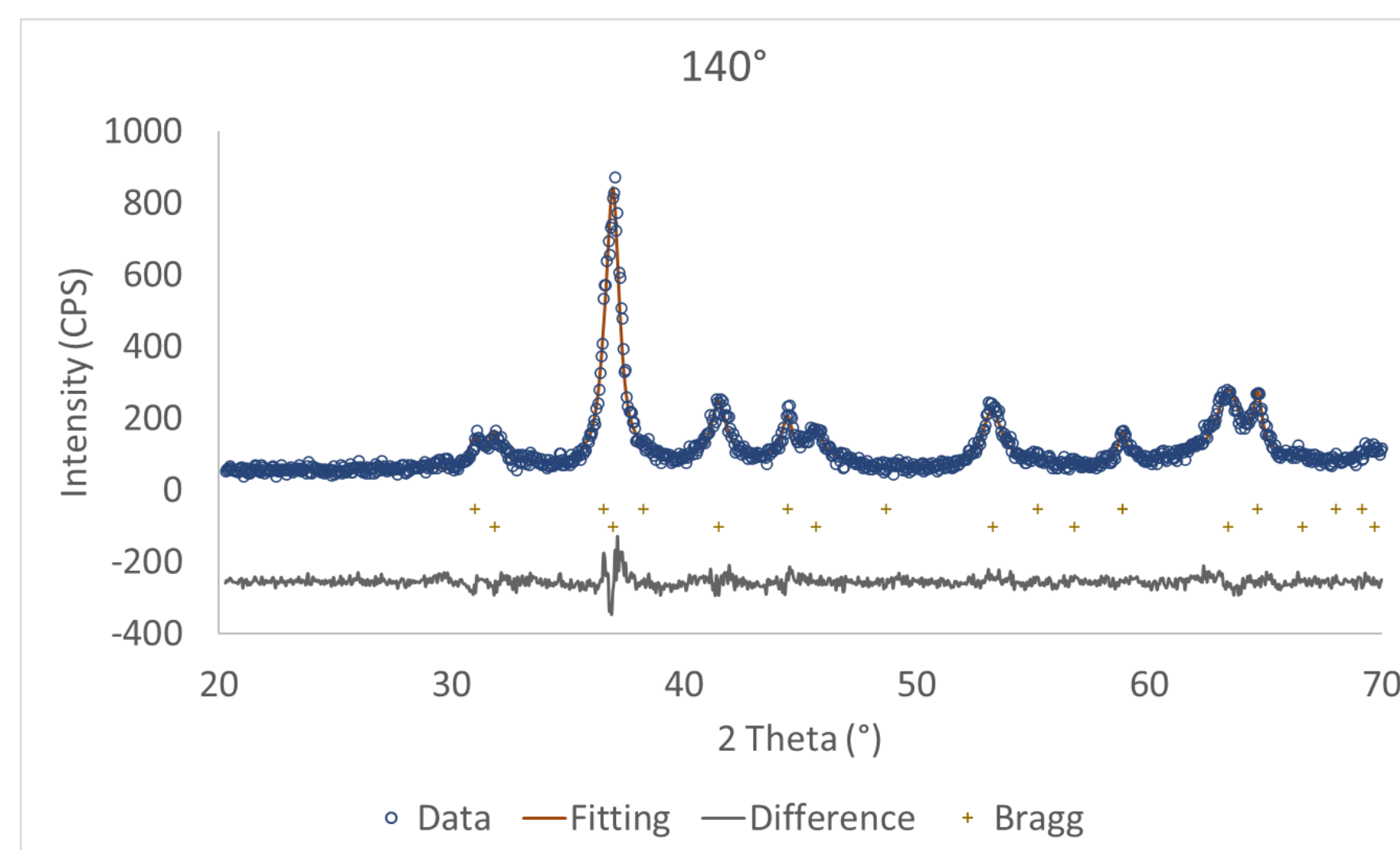


Figure 1: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 140°C

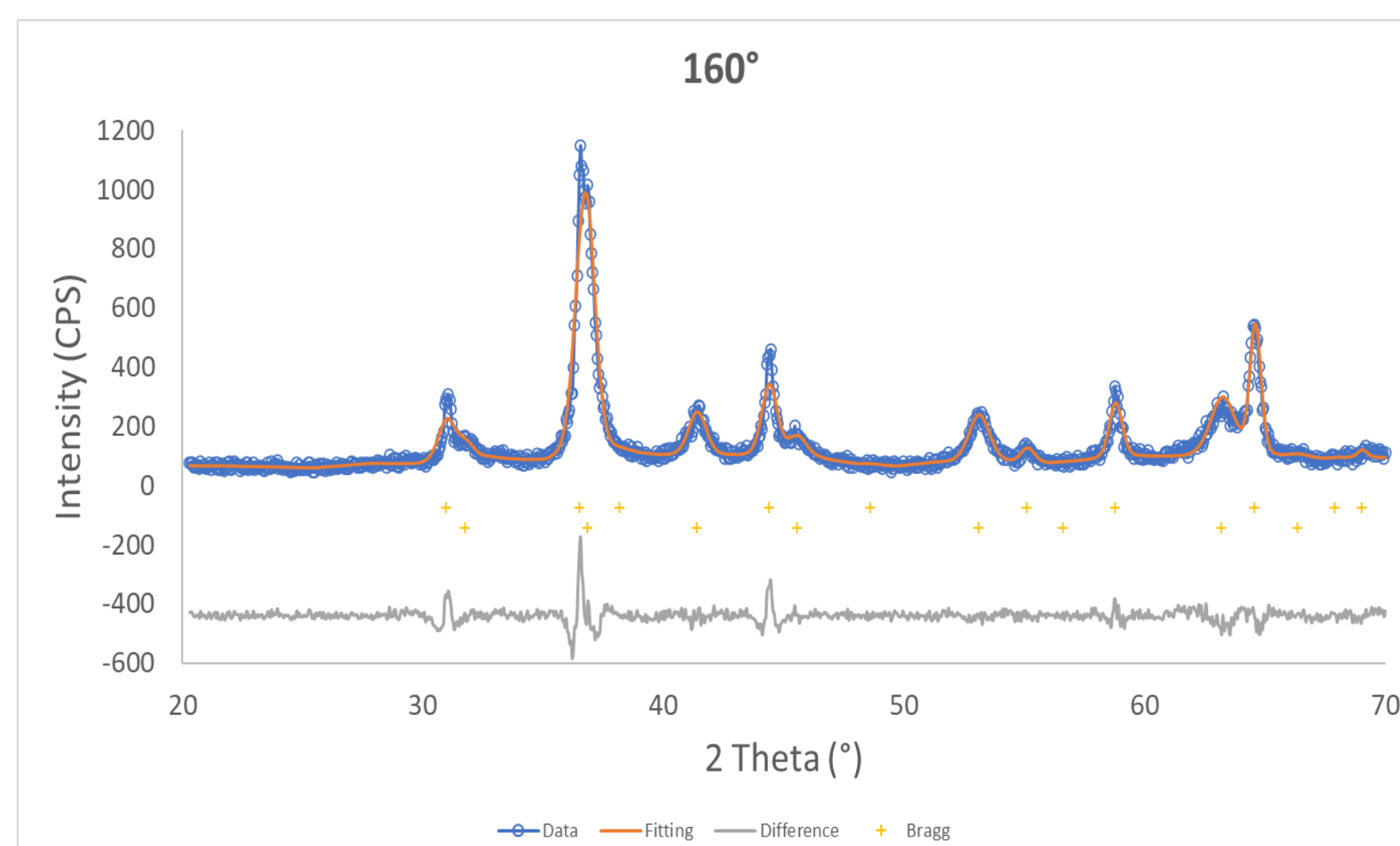


Figure 2: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 160°C

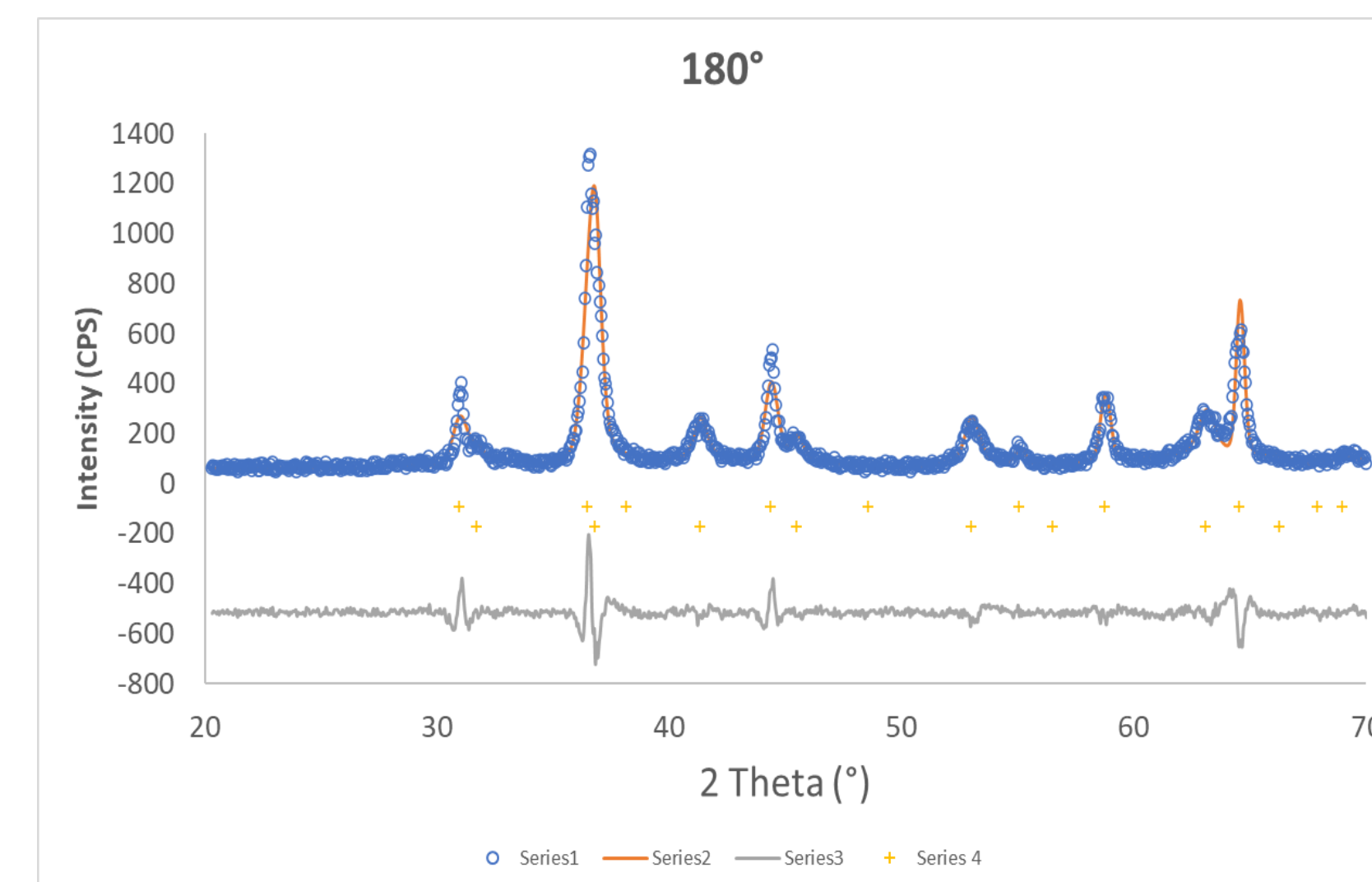


Figure 3: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 180°C

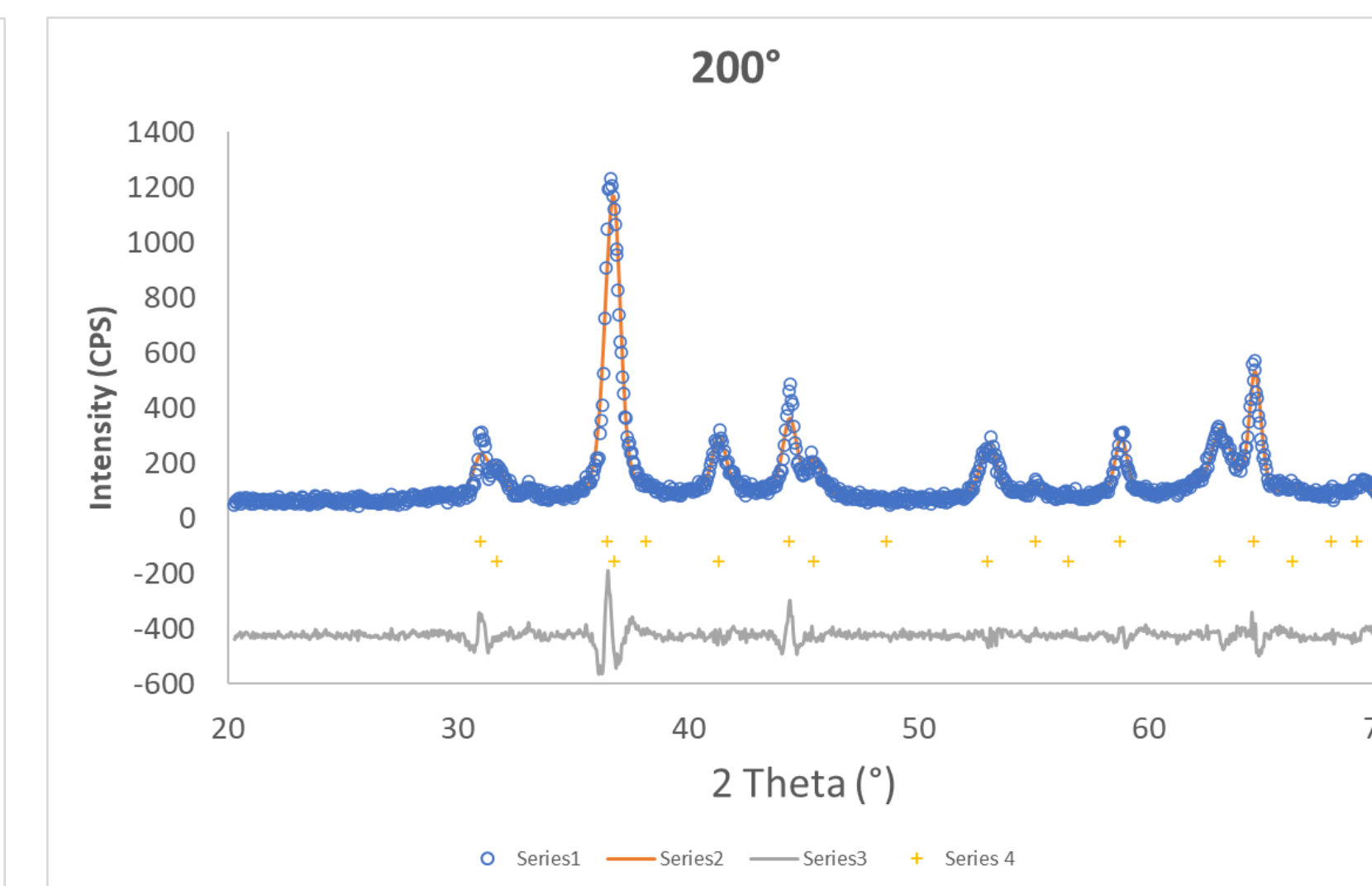


Figure 4: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 200°C

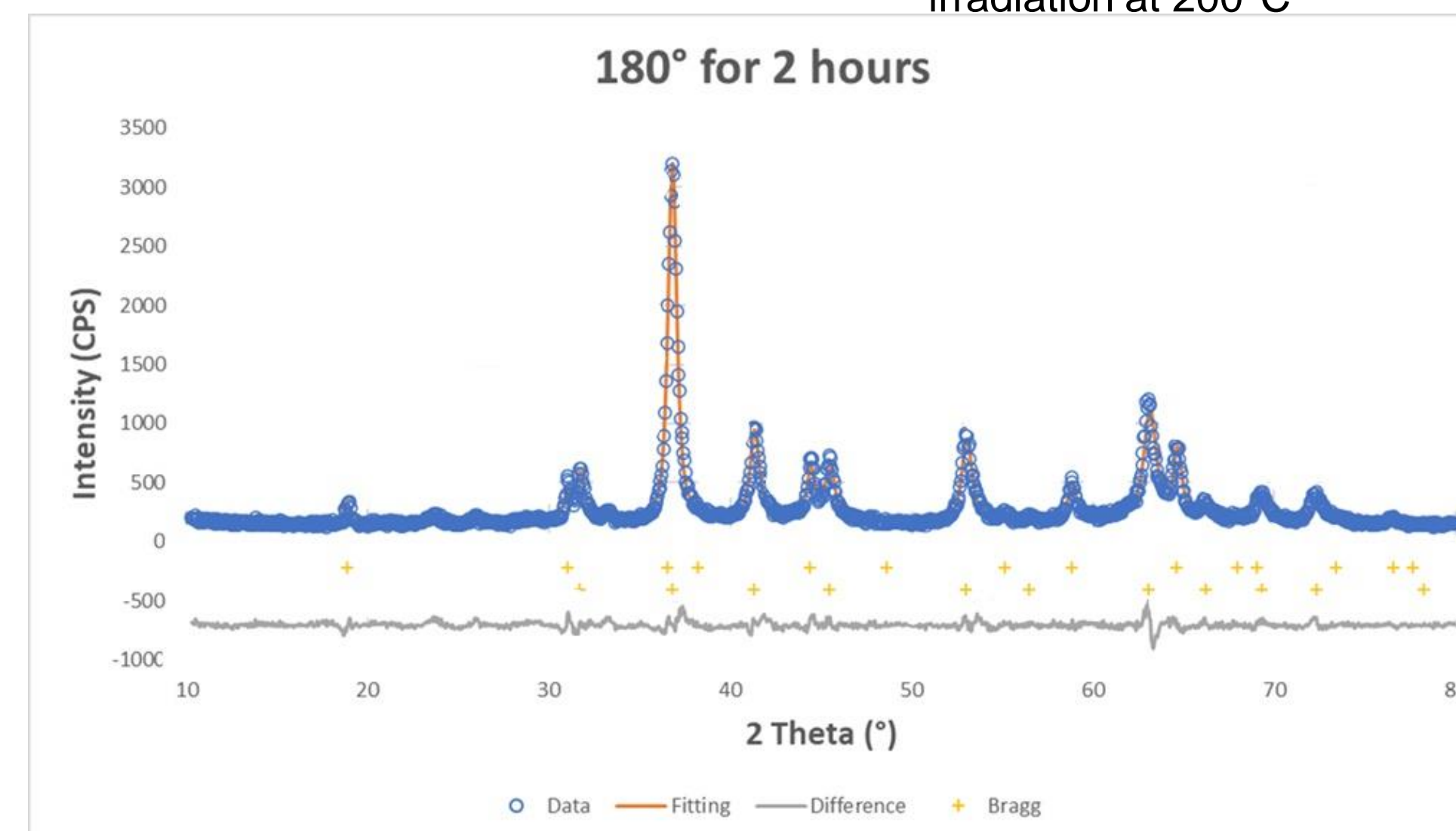


Figure 5: Fullprof fitting of Ni_xS_y synthesized under microwave irradiation at 180°C for 2 hours

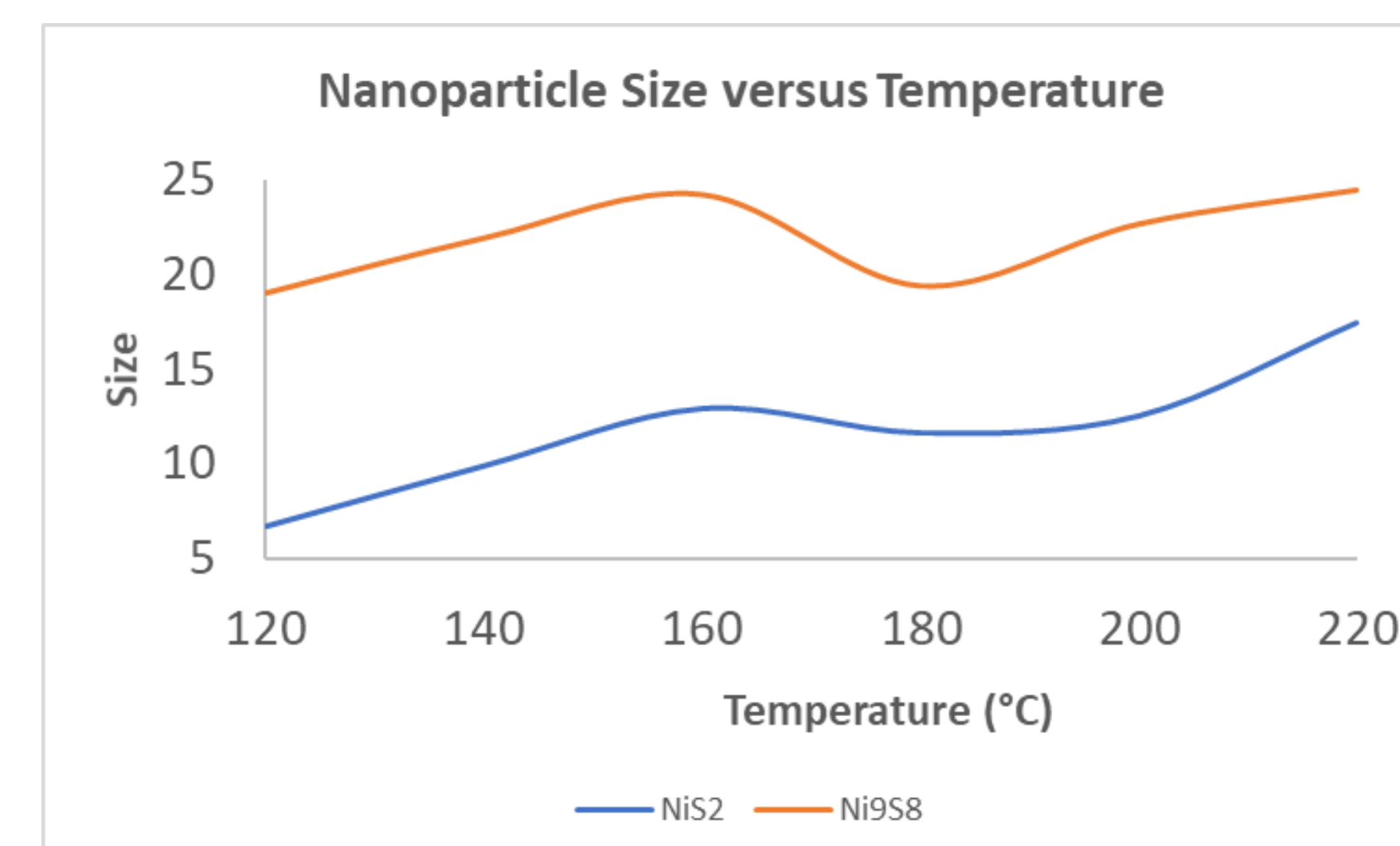


Figure 6: Effect of Temperature on particle Size for each Ni_xS_y Phase synthesized

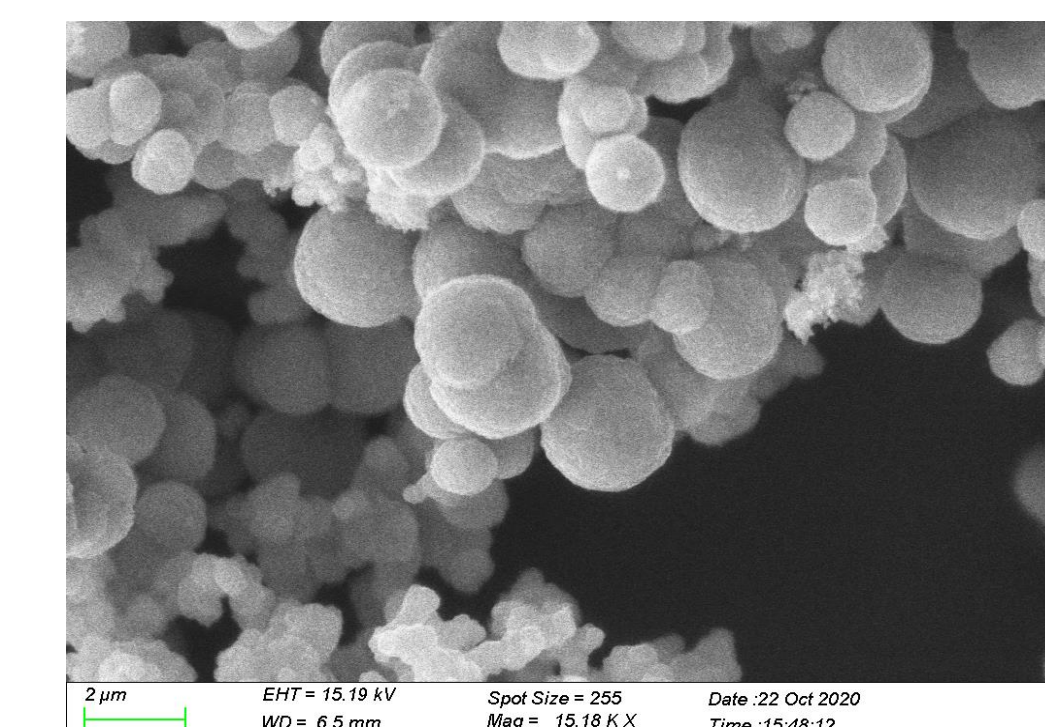
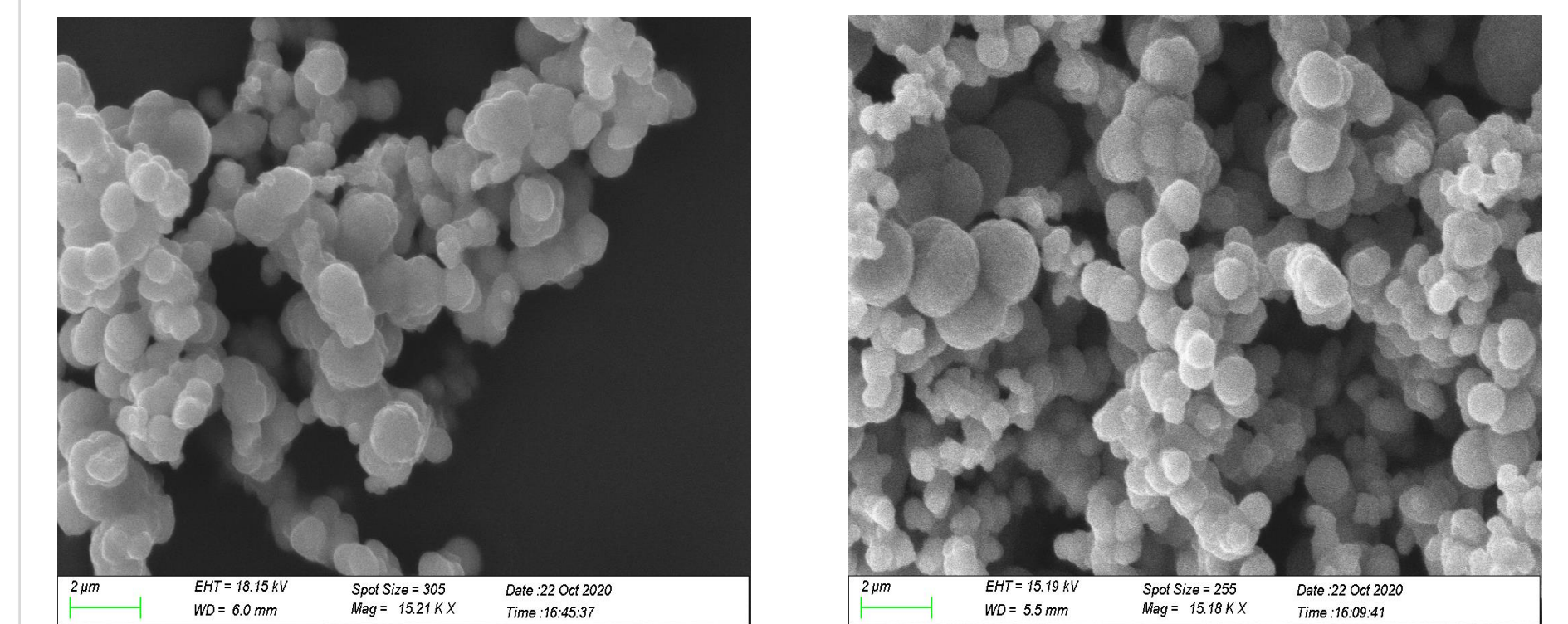


Figure 8: SEM of Ni_xS_y synthesized at 120°C (Top left) 160 °C (Top Right) and 200 (°C) bottom.

CONCLUSIONS

- Begin hold time after temperature is met
- Increase hold time
- 2 phase to 1 phase

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