# X-RAY DIFFRACTION ON THERMOELECTRIC SILICIDES AT HIGH PRESSURE

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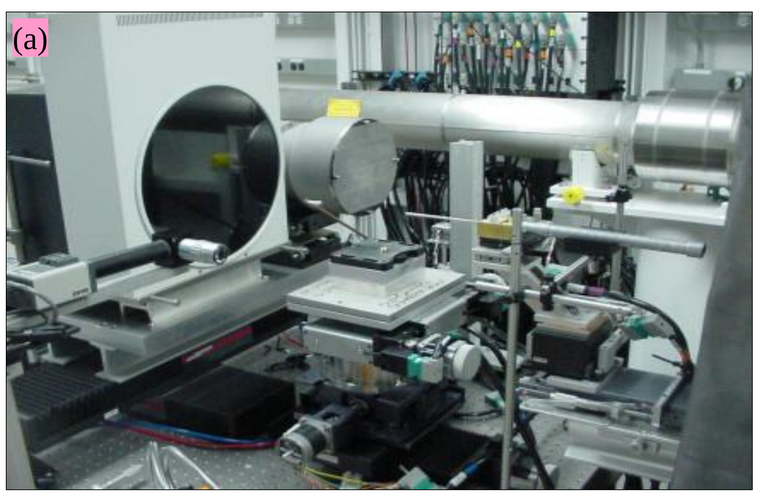
## ENERGY RESEARCH - THERMOELECTRICS

Cobalt Silicide ($CoSi_2$) is a transition metal disilicide that has gathered scientific interest due to its interesting thermoelectric properties and applications in silicon-based devices because of their high temperature stability. It has been reported that $CoSi_2$ undergoes a phase transition at around 0.4 GPa and again at 13 GPa [1]. Furthermore, at 13 GPa the material changes from a cubic cell to an orthorhombic cell, but details of the phase transition at 0.4 GPa could not be determined [1]. To further study the properties of $CoSi_2$ and understand its pressure induced phase changes, we recorded the structural behavior of $CoSi_2$ under pressure.

## EXPERIMENTAL DETAILS

X-ray diffraction experiments under high pressures were carried out at sector 16 IDB, HPCAT, Advanced photon source (Fig.1) and the incident photons had a wavelength of 0.4245 Angstroms. A diamond cell of Mao-Bell type with a culet diameter of 300 microns was used. The sample was bought from Alfa Aesar and was of 99.9% purity. The samples were loaded in a rhenium gasket preindented to 40 microns, with silicone fluid pressure transmitting medium and few ruby grains. Pressures were measured by the standard ruby fluorescence method.



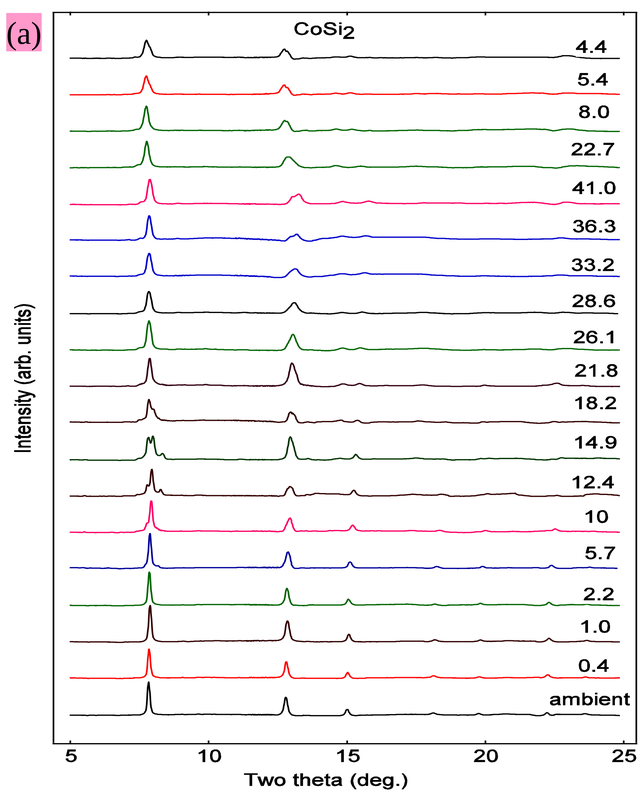


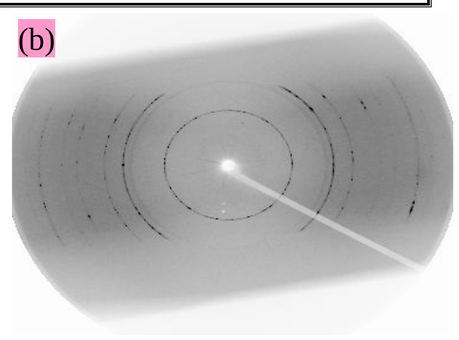
Diffraction images were collected using an imaging plate with an incident beam size of $20x20\mu m^2$. The typical exposure time for each pressure was 10-15 Sec. The images were then integrated using fit2d software, the background was fitted using PeakFit, the data was analyzed using both MDI Jade and Powder Cell, and then the analyzed data was graphed and fitted using Origin Pro.

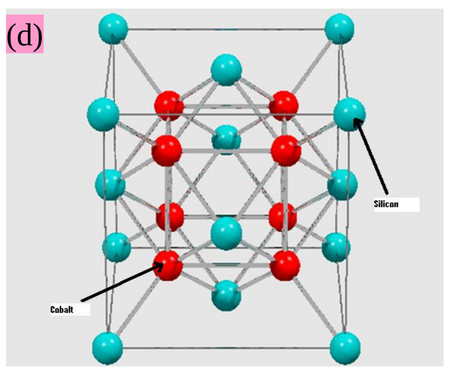
**Figure 1.** a. Experimental set up for high pressure x-ray diffraction at Sector 16 IDB, HPCAT, APS.
b. Mao-Bell type DAC

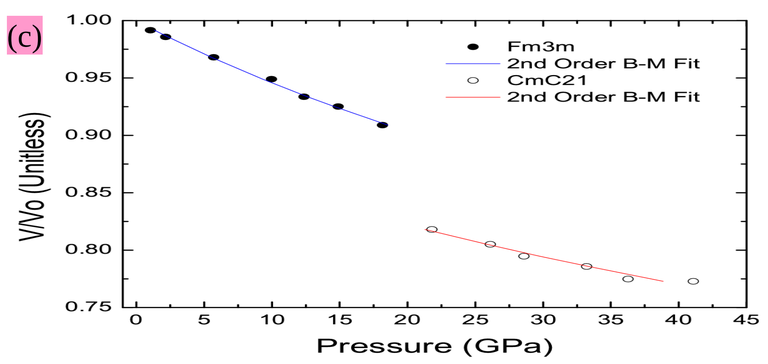
## DATA ANALYSIS

Using Jade we found that $CoSi_2$ is a cubic structure of space group Fm2m, with cell parameters of a=b=c= 5.35 Angstroms, at ambient pressure as shown in fig2 d. As the pressure was increased the volume clearly decreased, as expected.. There was also a phase change around 0.5 GPa, clearly highlighted by the fact that the peaks shifted a little to the left in fig2 a. The structure remained cubic but we could not pin down the exact space group. There was yet another phase transition at around 20 GPa as illustrated by the unifying of the leftmost peaks in the pattern corresponding to 21.6 GPa in fig2 a. We found that new phase was Orthorhombic and the space group was most likely CmC21. Fig3 c. shows the plot of Volume vs. Pressure.




The figure above shows the change in volume with a change in pressure. The dots show the actual data and the blue and red lines are second order Birch-Murnaghan equation of state fits for their corresponding phases. The gap in the middle represents the phase change and the volume collapse, which we found was about 10%. The compressive parameters in comparison with the bulk material are given in Table 1.

**Figure 2.** a. Representative x-ray diffraction patterns at different pressures for $CoSi_2$.
b. Raw data (before integration from Fit2D) of $CoSi_2$ at Ambient pressure.
c. Volume vs. Pressure graph of $CoSi_2$.
d. Structural diagram of $CoSi_2$ at ambient pressure.

**Table 1.** Compression parameters for $CoSi_2$

| | Phase 1 – Fm3m | Phase 2 – CmC21 |
|---|---|---|
| $B_0$ (GPa) | 161(5) | 197(31) |
| $B_0$' | 4 | 4 |
| $V_0$ (Å$^3$) | 155.26(28) | 139.0(22) |

## CONCLUSIONS

High pressure x-ray diffraction experiments on $CoSi_2$ were performed up to 41 GPa. There was an undetermined phase change at 0.4 GPa. Around 10 GPa, we saw the splitting of the major peak at 8 degrees. There was another phase transition at 18.2 GPa from a cubic structure to an orthorhombic structure of space group CmC21 evident from the recombination of the three peaks at 8 degrees. Our results confirm reports that under high pressure $CoSi_2$ changes from a cubic to an orthorhombic structure and that $CoSi_2$ is an unstable material under high pressure, however, our space group at the orthorhombic phase did not match the reported phase group and our volume collapse was massive compared to reported figures [1]. Furthermore, we were not able to gain additional insight into the structural changes at 0.4 GPa.

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## REFERENCES

1. Garg, A. and Vijayakumar, V., 2008., Pressure induced structural transitions in $CoSi_2$, *Journal of Physics: Condensed Matter*. 20, 395210.

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