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In-situ monitoring of phase transition by **XRD**

Monitoring phase transition of polycrystalline alloys in-situ is key to new process development and yield enhancement in applications such as source-drain contact formation, phase-change materials (PCM) and ferroelectric and piezoelectric devices.

Context

The manufacturing of microelectronic devices starts from material processing and engineering. Understanding what happens to materials in terms of their microstructure and phase during processing is of paramount importance. One example is the phase formation of silicides during rapid thermal annealing for ohmic contacts in photonic devices. How the final phase is formed with variable content of metal and semiconductor? What temperature is expected and at which heating rate is ideal to form that phase given a minimum thermal budget? These questions can be answered with in-situ XRD.

The challenge

The formation of ohmic contacts in microelectronics usually involves the diffusion of metal into one or more layers of semiconductor material during thermal annealing, typically, rapid thermal annealing (RTA). In a RTA process, temperature is risen in a very short period of time, usually within a minute. Monitoring phase transition during this very short time requires not only a good heating setup but also a very strong X-ray beam which can only be found at a synchrotron. At ESRF beamline BM05, fast acquisition and the very bright source available allow monitoring of phase transitions for several types of samples and annealing processes.

The results

Thanks to the very high brilliance of the X-ray beams available at the ESRF, phase transitions in polycrystalline alloys were smoothly observed in real process conditions. Several types of samples were studied with different metal concentrations in order to understand the impact of metal content in the phase transformation. A typical sample stack is shown in Fig 1. The study also aimed to determine the minimum thermal budget needed for the formation of ohmic contacts.

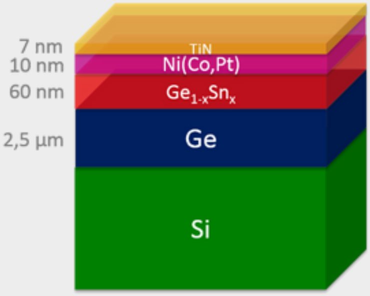
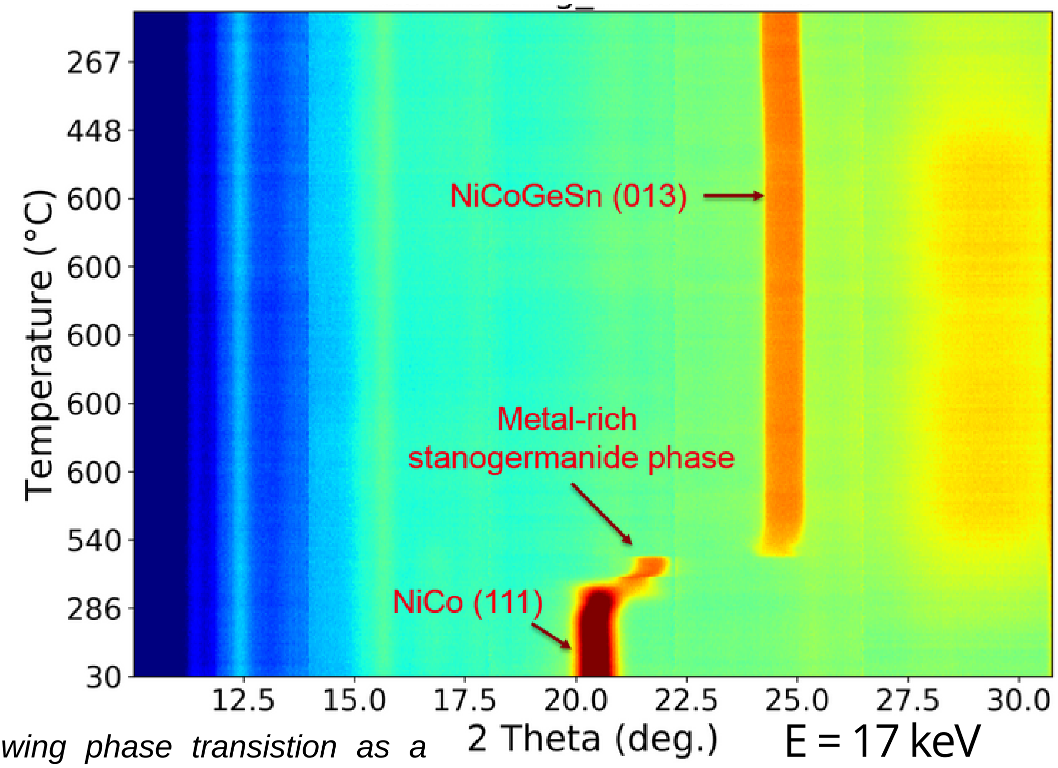


Fig 1. Illustration of typical sample stack used in this study with variable content of Sn and Co.



Picture showing phase transistion as a function of temperature for RTA method. All intermediary phases are clearly observed.

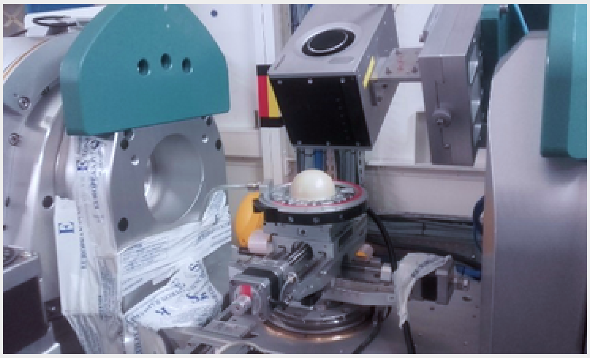
Conclusion

Phase transitions of the NiCoGeSn alloys are clearly identified during different types of annealing including RTA. Measurements could be performed for fast heatings going from room temperature to 600°C within 300 seconds. Using a lab X-ray source, 13 hours of data acquisition were required to perform the same measurement, revealing a different transition against temperature.

The difference observed is attributed to the low heating rate of the lab experiment. Slow measurement using a lab source is therefore not appropriate to study phase transitions under rapid thermal annealing process.

The technique

- A strong synchrotron beam at high energy is focused on a small spot which shines the sample area.
- A 2D detector is placed behind to collect diffraction data coming from the sample in a Bragg-Brentano geometry.
- Using the high energy beam and 2D detector available at ESRF allows for increased signal to noise ration and reduces measuring time.



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