X-Ray Photon Correlation Spectroscopy measurements of avalanches and hysteresis in Shape Memory Alloys

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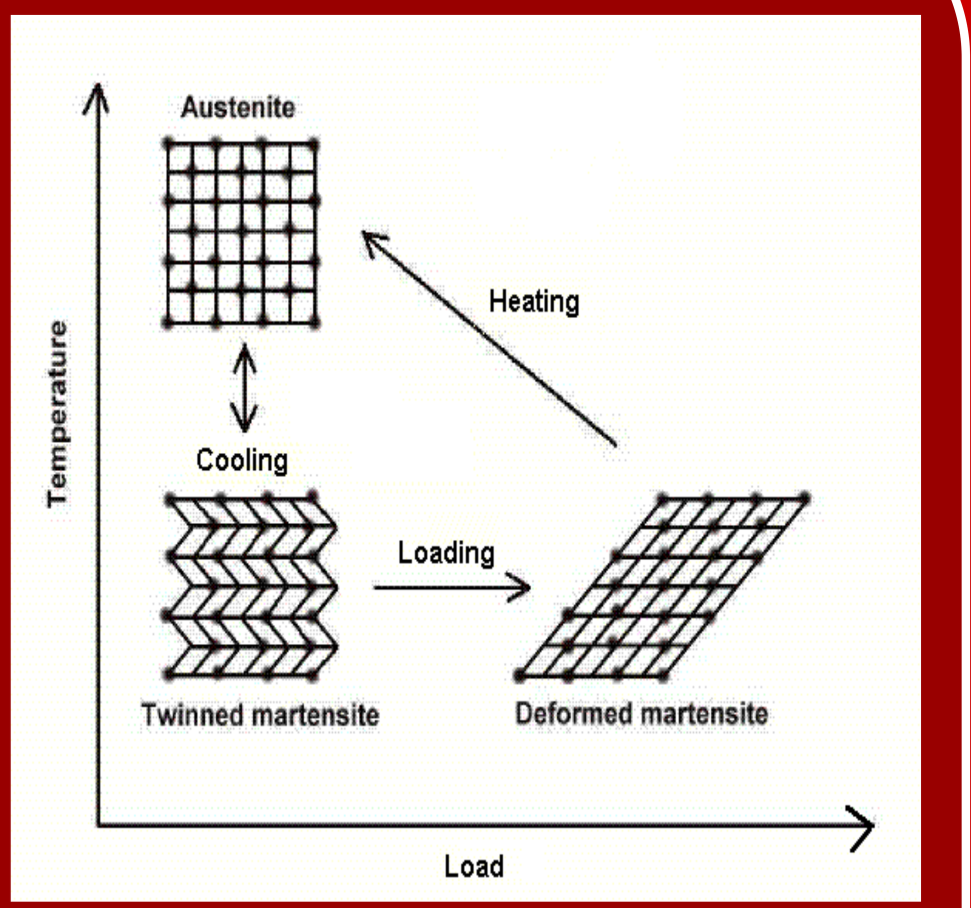
Introduction

A Shape Memory Alloy (SMA) is an alloy that has the ability to revert back to it's original shap. Once a SMA is strained, heating can return it to its previous configuration. This occurs due to a solid-to-solid phase transformation within the crystal structure of the metal in which atomic stacking layers are shifted.

While X-Ray Crystallography has been used extensively to characterize the different crystalline phases of SMAs, however the dynamical process that drive their transformation are still poorly understood.

We have used X-Ray Photon Correlation Spectroscopy (XPCS) to make in situ measurements of the transformation process of CuAlNi while it's subjected to temperature changes.

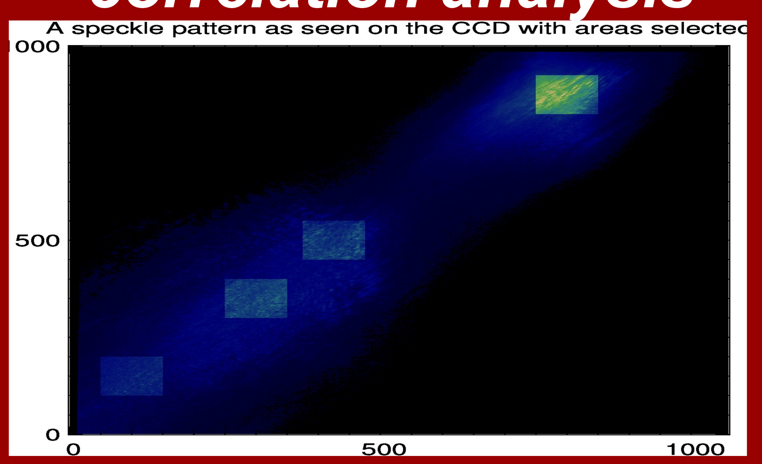
How does a SMA work?



Credit to: http://webdocs.cs.ualberta.ca/~database/MEMS/sma_mems/sma.html

Before the deformation, the crystal will be under the twinned martensite phase, by then deforming the metal we obtain the same phase, but simply deformed. The magic occurs when we heat the metal up, this is when the metal undergoes the solid-to-solid phase transition towards the Austenite phase. This is where the metal reverts back to the original shape. As the metal cools, it finally goes back to the martensite state, keeping it's original form and the cycle can be repeated.

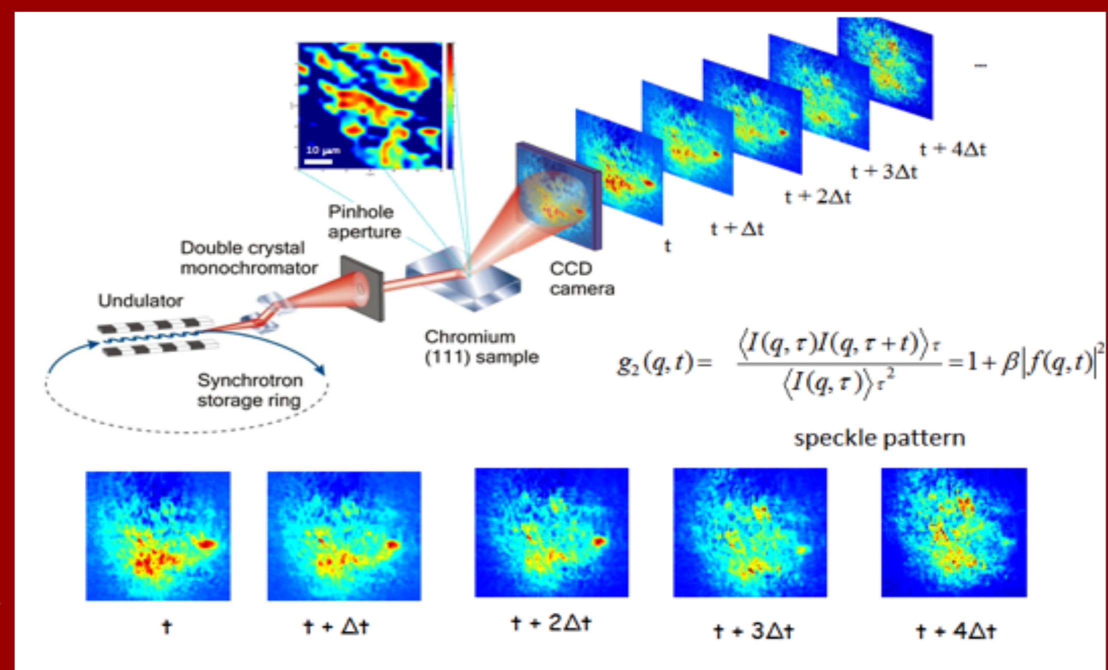
Methodology of the correlation analysis



The correlation method examines how much each frame looks like another and attributes it a number.We divide the CCD in multiple regions and use these to calculate the correlation between the frames. Thus, we obtain multiple regions in q-space for which we can plot the correlations.

What is X-Ray Photon Correlation Spectroscopy (XPCS)?

To find out about the dynamics of our system, we find a way of measuring it as it evolves through time. This is where XPCS comes in. Using a coherent monochromatic synchrotron source of X-Rays, we can irradiate our sample and look at the speckle pattern that it produces (i.e. How the X-rays diffract off of the crystal structure at any given point in time). The speckle pattern observed is essentially a picture of the imperfections of the crystal and constitutes a kind of fingerprint of the system at any given point in time. By then taking a time series of these speckle patterns, we can compare how this speckle pattern changes in time we can get a pretty good idea of how the system is evolving.

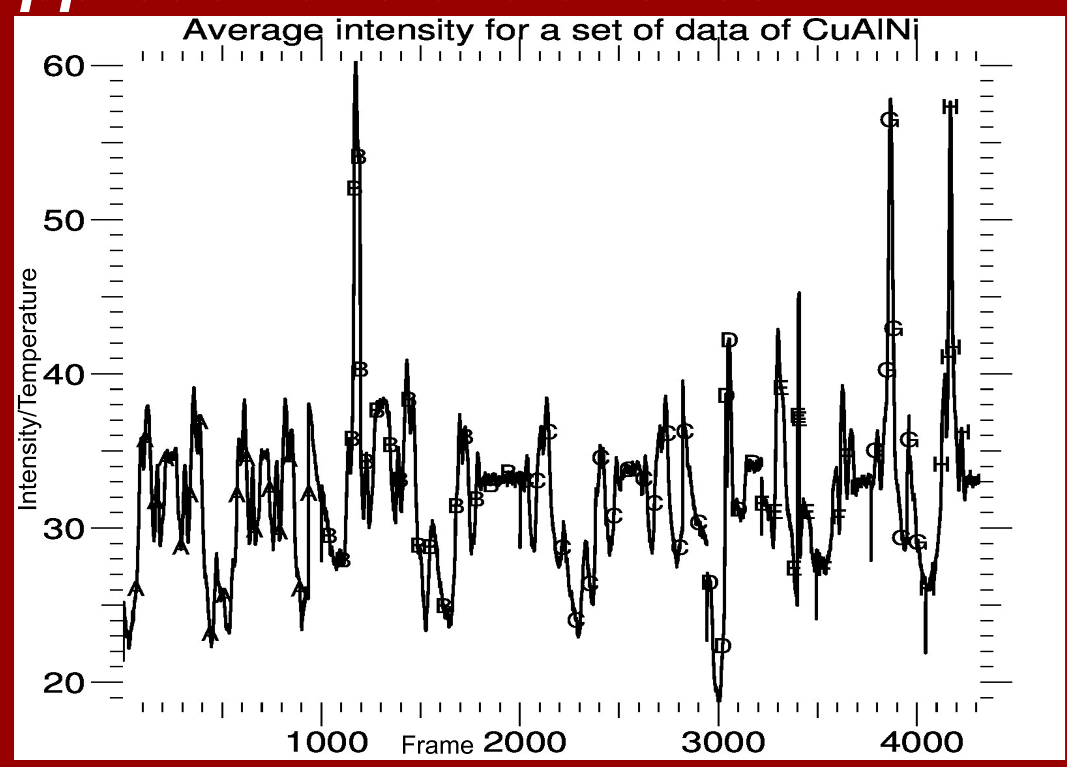


Schematic of X-ray Photon Correlation Spectroscopy measurements of antiferromagnetic domain fluctuations in Chromium

Credit to: http://oleg.ucsd.edu/speckle.htm#XPCS

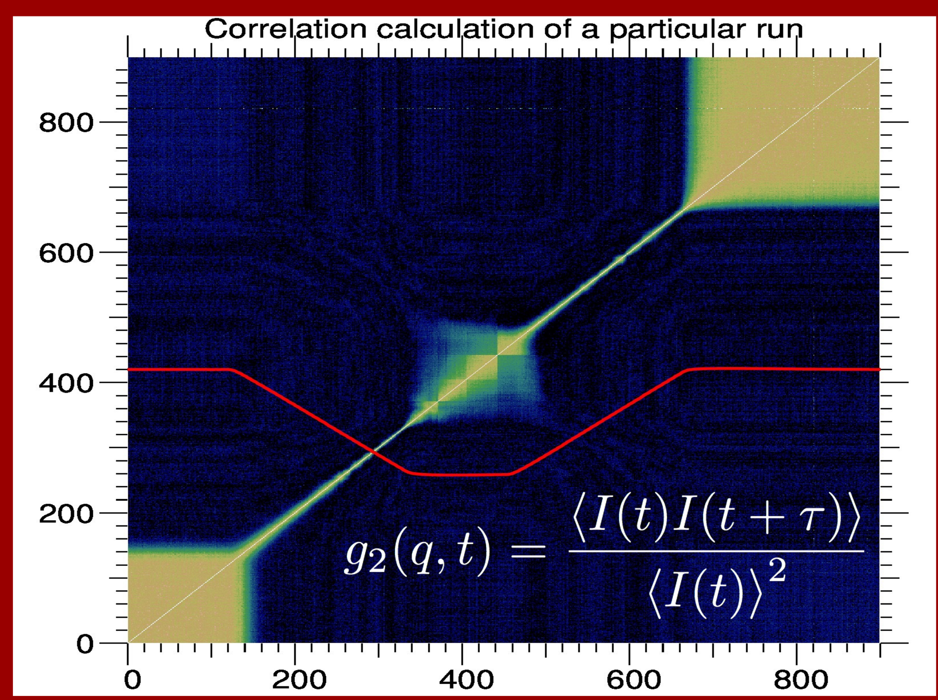
Average intensity approach and avalanches

There are multiple ways of then analyzing this type of data, one of them is to integrate and average the intensity recorded in each picture and looking at how it evolves in time as shown in the figure. As you can see, we start by observing a pretty regular and periodic pattern that coincides with the temperature cycles. Then it exhibits a jump and the behaviour changes. As the first cycles progressed, stress was building up in the crystal structure until a snapping point; this is the sudden disruption that we noted and we call these micro structural avalanches and can be noticed by this type of analysis, however it is not always clear from this picture when they occur.



Correlation approach and hysteresis effect

Here, we examine the correlation between each frame and seeing how they evolve through time. As we can see from the picture shown, it is now much easier to notice avalanches happen, notice the discontinuity process in the middle and the squares. We can see that it does not return to its exact previous state, since the first frames are almost not correlated to the final ones (for this particular run). This combined with what was observed in the average intensity approach allows us to say that a system is highly dependent on its previous history. This is what we call the hysteresis effect and is clear from the correlation analysis.



Conclusions

- The crystal transformation that a shape memory alloy undergoes does not for the most part happen via a continuous process but rather by discrete steps called microstructural avalanches
- X-Ray Photon Correlation Spectroscopy measurements have allowed us to conclude that the history of a material greatly affects what it will due in the future. This is the first time that the hysteresis effect has been observed at the scale of nanometers
- A material can be "trained" to go back to the same crystal structure by cycling the material multiple times through the same temperature quenches

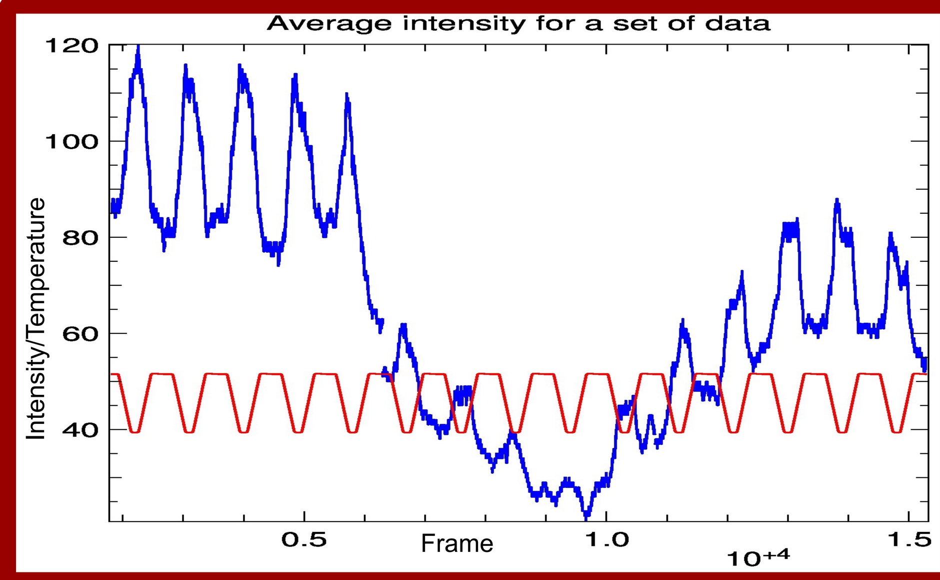
References and acknowledgements

Christopher Sanborn[1], Karl F. Ludwig[1], Michael C. Rogers[2], and Mark Sutton[2], "Direct Measurement of Microstructural Avalanches during the Martensitic Transition of Cobalt Using Coherent X-Ray Scattering," Phys. Rev. Lett. **107**, 015702 (2011).

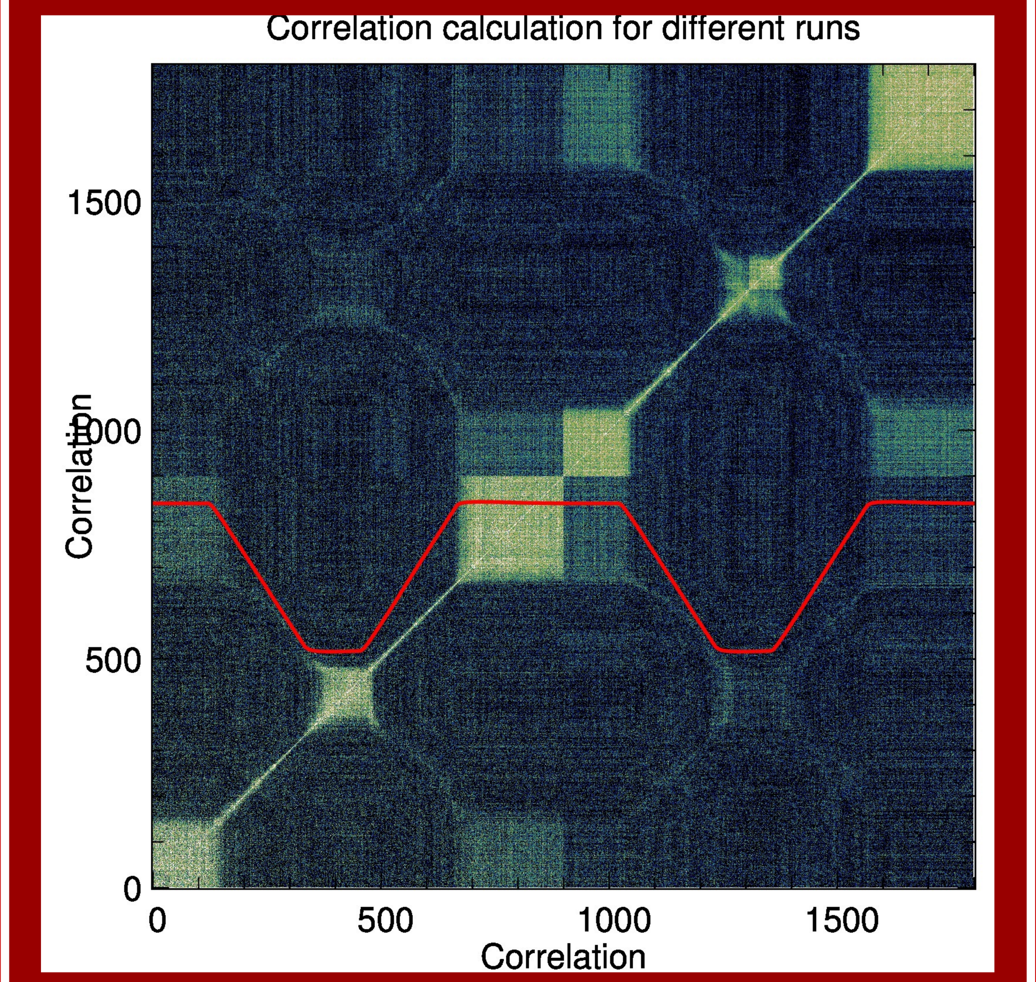
M. Sutton, S.G.J. Mochrie, T.Greytak, S.E. Nadler, L.E. Berman, G.A. Held and G.B. Stephenson, "Observation of speckle by diffraction with coherent X-Rays", *Nature* **352**, 608 - 610 (15 August 1991); doi:10.1038/352608a0

Special thanks to my supervisor, Dr. Michael Rogers without who this would've been impossible

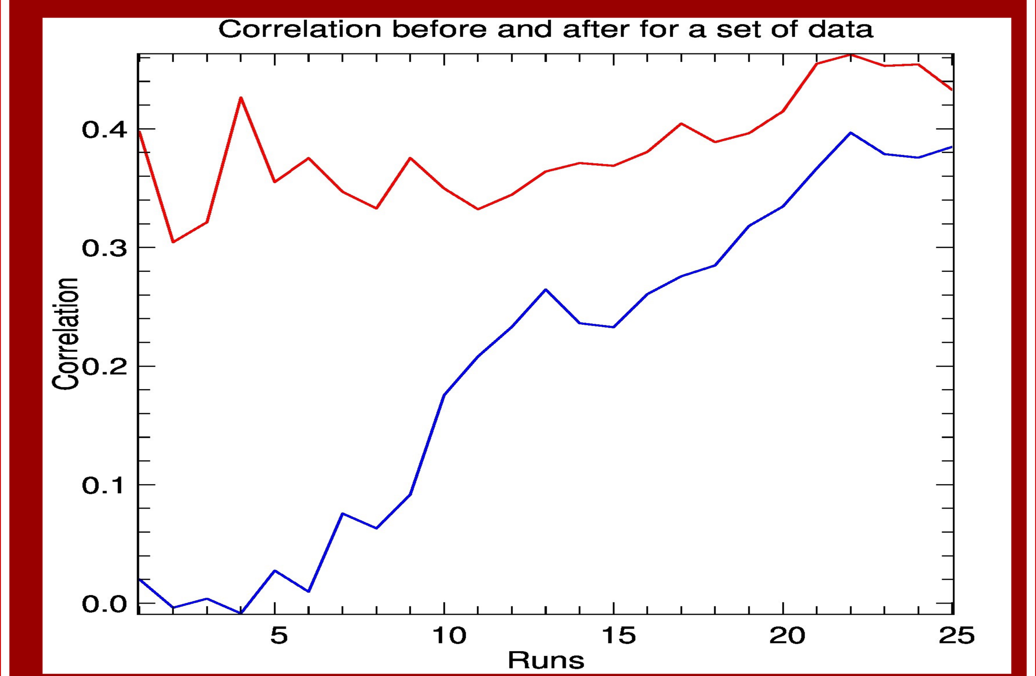
Some results



This is a particularly interesting set of data that underwent training. As we can see from the average intensity analysis, there is some semi periodicity, followed by a (clear) avalanche and then some recovery and an entrance into a new state, this motivated the further analysis of this run using correlation.



As we can see, the material doesn't seem to come back exactly as it was before. Also, we notice that the crystal seems to undergo an adjusting period in the plateau of temperatures. Multiple cross correlation calculations were performed for different runs.



Next, a more quantitative approach was taken and we took an average of the before and after correlations each run. As we can see from the graph above, where the correlation before is plotted in red and the correlation after in blue, we notice that the material seems to go back more and more to the same structure as we cycle the material through the same temperature cycles. We thus clearly observe the hysteresis effect and conclude that the material seems to get better and better at coming back to it's original shape, potentially tending towards some equilibrium