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Progress Report 1

Non destructive qualitative analysis of crystallinity[i] via X-ray diffraction measurements

Simone Napolitano
Mentor: *Riccardo DeSalvo*

My project deals with checking the validity of a non-destructive quantitative analysis via X-ray diffraction. My mentor and other SURF students have developed this technique last year, in order to control the quality of mechanical components for Advanced LIGO[ii]. This new technique may allow determination of volume fraction to a precision of the order of 1% for highly amorphous sample (>85% amorphous phase volume), and a precision of the order of 5% for samples with lower amorphous phase volume.

Thus this technique will let us calculate the amount of crystallinity in very amorphous materials as the glassy metals.

A crystalline contamination compromises the extraordinary properties of the alloys themselves.

In Keck laboratories LIGO group is researching a potential material for flex-joints in mirror suspensions for Advanced LIGO[iii].

I've been shown the use of splat quencher and arc melter and I'm working with the mini arc melter for the material production of MoRuB [one of the possible materials for flex-joints in mirror suspensions]. Using stoichiometric calculations we get the right amount of each pure element to use. We get Molybdenum ingot from the pure element in powder. The weighted Molybdenum gives the amount of Boron and Ruthenium. The pure elements are put in the mini arc melter in which is created an inert atmosphere through 3 cycles of vacuum [via mechanical pump] and Argon. We put a ball of Titanium inside the melting room; if this latter is not clean the Titanium [melted for at least 1 minute] looses its characteristic silver brightness. The elements are melted twice [the room is opened and cleaned each time] and then finally sucked and casted in rectangular ingots. These latter are cut and weighted in amount of 0.135 g each, remelted; we finally have small balls of MoRuB alloy. The little balls are eventually sent to the splat quencher. The ball is put in the middle of a Copper coil, a electromagnetic field is generated and this latter opposed to gravity, makes the ball levitate. The heat dissipated by the coil melts the ball, and laser detector actives 2 Copper pistons when a drop of alloy falls down.

During these first three weeks I had laser safety training, X-rays safety training and using of machine training always with X-rays.

In order to check the validity of the new technique, I must collect data on the same samples both via x-rays and differential scanning calorimetry. DSC is a well-known calorimetric analysis that allows to determinate the thermal transitions and the related [if exiting] heats of transitions in a material. Through this data it is possible to determinate many properties of the sample, e.g. crystallinity.

First step of my work has been searching a suitable alloy for my project. The alloy I need has to present thermal transitions at relative low temperatures [best is under 873 K, normal limit for traditional DSC], thermal transitions have to occur in one peak only [more peaks generate higher errors in data analysis], and exothermic relaxation at T_g has to be small [I can neglect this term for it is usually 2 orders lower than ΔH_x]. I could use a normal DSC and not a Modulated DSC [in which a software separates reversible and irreversible signals], even if I would like to try to use the latter.

I've checked in books and articles DSC curves of amorphous alloys and finally found a "good" alloy: $Pd_{43}Ni_{10}Cu_{27}P_{20}$. In a normal thermal DSC curve [under 873 K] of this alloy to a glass transition follows a one-only peak crystallization and a one-only peak of melting.

Thus, I can suppose that the integrated area of the crystallization peak gives a measure of total amorphous volume.

Prof. Johnson, a pioneer in glass metals studies, suggested me to meet Chris Veazy, a researcher working at Materials Science Dept. at Beck laboratories who produced that alloy and wrote same papers on it. Chris gave me a sample of its alloy.

I began to use the X-ray machine to collect data on the total amorphous alloy sample and have a DSC of it.

In this month I'll collect data on different samples of PdNiCuP alloy with different amount of cristallinity.

To do this I can

1) produce the alloy itself and cast it in a particular triangular shape, the thickest [top] part should be totally crystalline while the bottom [minor thickness] should be totally amorphous and thus I would have a gradient of crystallinity running through my sample. I'll finally get my slices cutting my cast sample in parallel planes to the base.

2) produce a totally amorphous alloy and reheat slices of this latter at different temperatures for different time programs, in order to introduce crystallites into the sample [higher temperatures and longer program periods should provoke greater crystalline contaminations] and get samples with different cristallinity.

So doing I'll be able to use DSC as a standard method to verify our new technique, and have an idea of the limitation of both analyses

I'll analyze my data collected via X-ray with the new technique [using a software named Kaleidagraph] and via DSC in 2 different methods:

1) Amorphous Volume Fraction via ΔH_x analysis

2) Crystalline Volume Fraction via ΔH_x and ΔH_m.

The second approach will require maybe the use of a High Thermal DSC.

A third method could be introduced, theoretically I could even analyze Δc_p induced by the observed glass transition, but practically these variations usually are so smaller that errors inducted by the same analysis would compromise the final measure.

Anyway using DSC as a standard method to verify our new technique, and having an idea of the limitation of both analyses will be my goal for next month.

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[i] In my work I'll use, for the sake of simplicity, the term *crystallinity* instead of the more appropriate expression *crystalline fraction*

[ii] Emmerson, Brian (2002). X-ray scattering measurements of crystallite contamination in glassy metals (updated). Presentation, LIGO document LIGO-G-020444-00-R

[iii] 1) DeSalvo, Riccardo (2002). Are Glassy Metal flex joints better than fused silica fibers for mirror suspensions? Presentation, LIGO document LIGO-G020445-00-R

2) Simoni, Barbara (2002). SURF final presentation, Phase transition heat in MoRuB. Presentation, LIGO document LIGO-G020439-00-R

3) Mantovani, Maddelena (2002). SURF final presentation, Hardness and Elasticity Measurements in MoRuB. Presentation, LIGO document LIGO-G020440-00-R

4) Tirelli, Stefano (2002). SURF final presentation, Stress-strain behavior of MoRuB glassy metals. Presentation, LIGO document LIGO-G020441-00-R

5) Hall, Michael (2002). SURF final presentation, Physical Property Measurements of Glassy Metals. Presentation, LIGO document LIGO-G020443-00-R