ION IRRADIATION-INDUCED NANO-CRYSTALLIZATION METALLIC GLASSES (AMORPHOUS METAL)

A Senior Scholars Thesis

by

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ABSTRACT

Ion Irradiation-Induced Nano-Crystallization of Metallic Glasses (May 2013)

May 2013

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This project idea is proposed in order to develop the understanding of the mechanisms responsible for Nano-crystal phase formation when metallic glasses (amorphous) is exposed under the high energy ion irradiation and is quantified the resulting effects on the material behavior. The objectives of this project are to: (1) quantify the response of metallic glasses to ion irradiation, specifically, damage cascade formation and subsequent structural evolutions along ion tracks, (2) identify the mechanisms responsible for ion irradiation-induced Nano-crystallization, specifically, the paths of crystal nucleation and growth, and effects of energy deposition rate and subsequent energy dissipation rate on crystal formation, (3) quantify the resulting crystal phases, densities and distribution under different ion irradiation conditions and identify the governing factors to reach controllable crystal formation, and (4) measure the hardness elastic modulus and ductility of the metallic glasses containing Nano-crystals created by ion irradiation, and quantify shear band interactions with crystal of different sizes, shapes and matrix distribution.

It is expected that this study will contribute to new fundamental understanding of mechanisms responsible for the creation of Nano-crystals in metallic glasses by ion irradiation, and the role of the altered structure in the modification of the materials mechanical response. It will impact fundamental understanding in several materials science subfields. In the field of ion-solid interactions, phase transitions from amorphous to crystalline in metallic materials are poorly understood. This study will contribute to a more complete understanding of the structural evolution occurring over different time scales involving damage cascade creation, thermal spike formation, and structural relaxation. The roles of excess free volume and thermal energy deposition on the mechanisms responsible for Nano-crystal phase formation will be identified. In the field of Nano-mechanical behavior of materials, this study will identify the roles of Nano-crystals on the mechanical response of metallic glasses. It will further contribute to our understanding of shear band nucleation and propagation, and deflection and attenuation by Nano-crystals of different mechanical strengths and interaction cross sections.

DEDICATION

I would like to dedicate this thesis to my beloved parents who are currently living in Vietnam, Mr. Hong Tran and Mrs. Tuyet Nguyen. There is no doubt that without their encouragement and supports, I would never have completed this process.

Besides, I would like to have special thanks to Dr. Shao, and Mr. Robert B Balerio who are helped me to finish this research project. Moreover, I will not discover the knowledge about the metallic glasses (amorphous metal), if I do not have a help and guide from Dr. Shao.

ACKNOWLEDGMENTS

I would like to acknowledge Dr. Lin Shao for his supports and guidance throughout the research. Also, I would like to acknowledge Mr. Robert B Balerio, my co-worker in this study for guiding me how to indent metallic glasses.

Besides, I would like to acknowledge Dr. Wilson Serem who trains me to use the Hysitron Triboindenter Nanoindenter (341-F).

CHAPTER I

INTRODUCTION

Metallic glasses (MGs) are amorphous alloys without any type of crystalline order structure. Metallic alloy (Figure 1) is lacking long-range periodicity of the atomic arrangement. This concept can be understand as cooling the liquid alloy below its thermodynamic melting point and then it's failing to crystallize but it will solidify. In order to solidify without crystallizing, the metallic glasses have been cooled down at the high rate to suppress the nucleation and growth of crystalline phase. They are formed by the process of rapid quenching of molten alloys with a high cooling rate (~ 10^6 K/s to avoid the appearance of crystallization in material – studied in early 1960s). [1]-[4].

There is an obstacle to produce the metallic glasses because of the extremely high critical cooling rate to form amorphous structure in the alloy. This cooling process is limited the achievable size of forming metallic glasses. Since the 1980s, the significant progress had been made in order to form the liquid metal with significant lower cooling rate. The metallic glasses had been modified the composition, so that the liquid metal increase its viscosities. [5] At the present time, the bulk metallic glasses with large diameter have been fabricated [6]-[7].

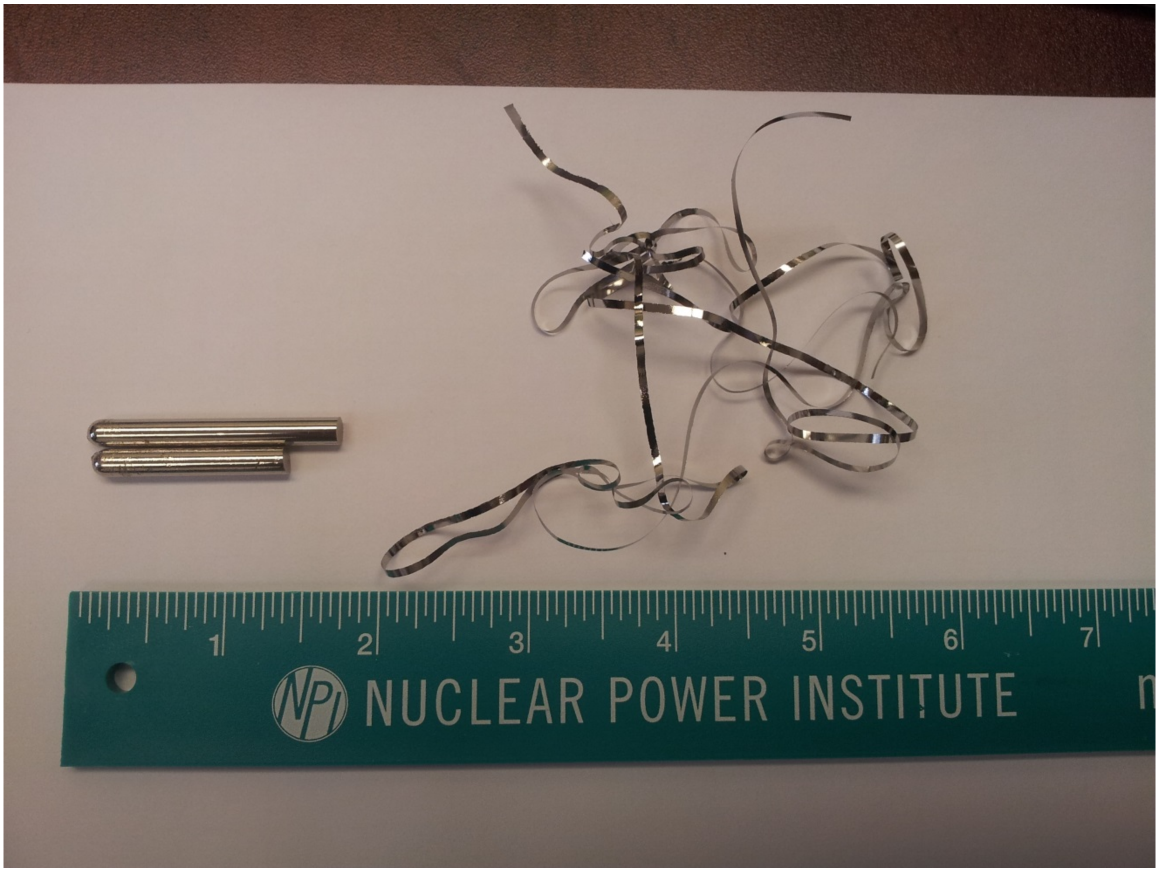

Figure 1: Metallic Glasses

Metallic glasses (Figure 1) have eliminated their grain boundaries and crystalline structure, so this process makes the structure of the amorphous metal become more useful and generate much interest in studying their mechanical properties. The metallic glasses have built strong resistance to wear and corrosion, and this is a unique result in their mechanical and chemical properties. MGs will have high tensile strength, large elastic region, and good resistance to fatigue. [8]-[11] However, the drawback of limitations of MGs is the low ductility at room temperature. The lack of ductility in the metallic glasses will be show clearly under uniaxial compression/tension.

Figure 2: Scanning electron micrograph of brittle MGs failed after tension

The shear bands will be developed when the MGs are applying load and the regions of highly strain localization is developed as well. With the small crack in the material structure (Figure 2), the MGs will quickly propagate through the entire material and this will lead to the result failure in whole structure of metallic glasses.

In order to increase the ductility properties in amorphous metal, the introduction of the nano-scale structure can arrest the shear band which lead to the failure. By forming the nano-crystal in the metallic glasses, the ductility properties can enhance significantly. The shear band which causes the failure in the structure can be trap by the nano-crystal, and the stress energy can be release by this nano-crytal as well. [12] By proving the improvement in the mechanical properties, the MGs can survive the uniaxial compression/tension, and the partial crystallization of MGs can be achieved by bending, indentation, annealing, irradiation with electrons, mechanical alloying, roll-forming of powders, sintering or compaction.

CHAPTER II

METHOD

Nano-indentation test

For basic investigation to metallic glasses, the plasticity properties can be reflected to the shear band formation and propagation. Nano-indentation is particularly well-suited for this study about shear band formation and propagation. Nano-indenters have high force and depth resolution which make verifying shear band nucleation and propagation more accuracy. The observations about discontinuities in load versus depth response have been shown to correspond to the nucleation of shear band with the individual resolution events. [17], [18]. Nano-indention studies of amorphous have included measurement of hardness and elastic modulus as a function of depth [19]. Besides, the effect of loading rate on the transition from localized to homogenous plastic deformation, and the formation of Nano-crystals near shear bands [20].

Specimen preparation

The metallic glasses were provided by Dr. Shao who is an advisor for this research project. The MGs specimen preparation is produced by using melting-spinning technique. After being melted, the high purity metals will be gone through Ar atmosphere and then injected on a large rotating wheel. The rotating wheel technique will help cool the MGs internally by using water or other liquid as a cooling material. The rotation frequency of rotating wheel and thermal conductivity of water or other fluid will affect the cooling rate. The achievable cooling rate is required staying in the order of 10^4-10^7 K/s range. The fabricate specimens are typically produced in a range of few millimeters in width and tens of microns in thickness. In this research project, we will pay attention in Zr and Cu based MGs such as $Zr_{55}Cu_{30}Al_{10}Ni_5$ and $Cu_{50}Zr_{45}Ti_5$ alloys. The reason to choose studying these alloys is based on previous study, and these alloys are well studied with massive available literature on crystallization. Moreover, $Zr_{55}Cu_{30}Al_{10}Ni_5$ and $Cu_{50}Zr_{45}Ti_5$ alloys also exhibit high mechanical strength in range of 1700-2160 MPa.

Experiment approach

High energy ion-irradiation

High energy ion-irradiation (Figure 3) is a method to choose studying about a phase change of amorphous metal as a function of ion penetration depth. Due to high energy ion-irradiation, there is a momentum transfer of ion which will create a chain interaction in a forward direction of ion. This phenomenon will be reflected in the distribution of displaced atoms and free volume. If displaced atoms and free volume (empty sites) recombination in a similar way as interstitial-vacancy recombination in crystalline material, we can predict a results of this experiment toward metallic glasses. There will be a high volume of free volume created near the surface, and massive density damage cascade at larger distance to the surface. In order to generate the governing factors in defining crystalline phase nucleation, the crystal depth distribution could be observed through cross-sectional TEM.

Figure 3: High Energy Accelerator

Ion-irradiation at elevated temperature

The specimen will be heated to a temperature which is lower than the MGs glass transition temperature, so that the temperature gradient between ion track region and the matrix will be reduced. At this temperature, the specimen will be ion-irradiated, so that the cooling rate of the thermal spike region will be possibly reduced to a value less than MGs critical cooling rate. Therefore, crystallization of ion track should be appeared. If the crystallization can be picked up, we can expect there is a formation of nano-crystals along with the ion track (Figure 4). Due to the ion-solid interaction phenomenon, the chain crystal distribution will diminish at the end of the projected range. By creating nano-crystal inside of MGs, the mechanism properties of amorphous will be increase significant such as the ductility of metallic glasses. The crack will be trapped by the nano-crystal formation on the surface of the MGs.

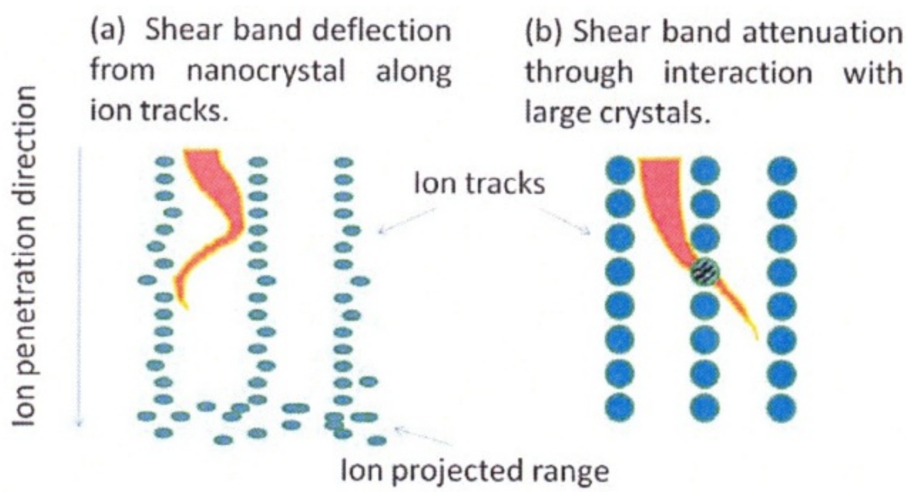

Figure 4: Crystals are formed by ion-irradiation

Cluster ion-irradiation

Ion-irradiation would be performed with the experiment of cluster ion-irradiation, rather than mono-ion. The cluster ions term was introduced to get an approximation of multiple ion tracks on the matrix. There will be a damage cascade creating by each individual atom with same clusters. The energy deposition will be higher correlation to the large damage cascade of the cluster. With the center point of the cluster ion damage cascade, the energy dissipation rate will be reduced because the thermal gradients around the ion track were reduced. Thus, the crystallization of damage cascade region can be achieved.

CHAPTER III

RESULTS

Although there is a limit about the time for this project, we still can initial achieve an expected results as a planning approach. We did get a solid result for the first approach High Energy Ion-irradiation.

Upon the experiment with high energy ion-irradiation, the atoms are displaced from their original locations to a new position where has a deeper location in the material. With the experiment of ion-irradiation, there is a nature of ion interaction between ion with the material to create a displacement of atoms and a free volume. This collision between ion and material can be treated as a pair of "interstitial and vacancy" defects. The recombination of free volume and displaced atoms can be achieved when those defects are in close to each other. Boltzmann Transport Equation and Monte Carlo method will be used to calculate the depth distribution of free volume and displace atoms in MGs. The recombination phenomenon of "interstitial and vacancy" will correspond to the concentration of imbalance in the number of free volume and displace atoms. This experiment also correlates to a concept of structural relaxation. During this experiment, I have a support from Mr. Balerio who provide me the calculated free volume and displaced atoms profile in Cu, which is caused by 3 MeV Cu ions (Figure 5). This result related to his Ph.D. thesis. Besides, I have a support from Dr. Shao to come up with the "defect" imbalance calculated and plotted in Figure 6. Figure 5 was part from his research project about metallic glasses. The results showed that the net free volume is negative at the end of the projected range while the positive results can be observed near a surface of material. Therefore, there are two different mechanism drive atomic mobility in two regions (free volume near surface and temperature caused by thermal spike).

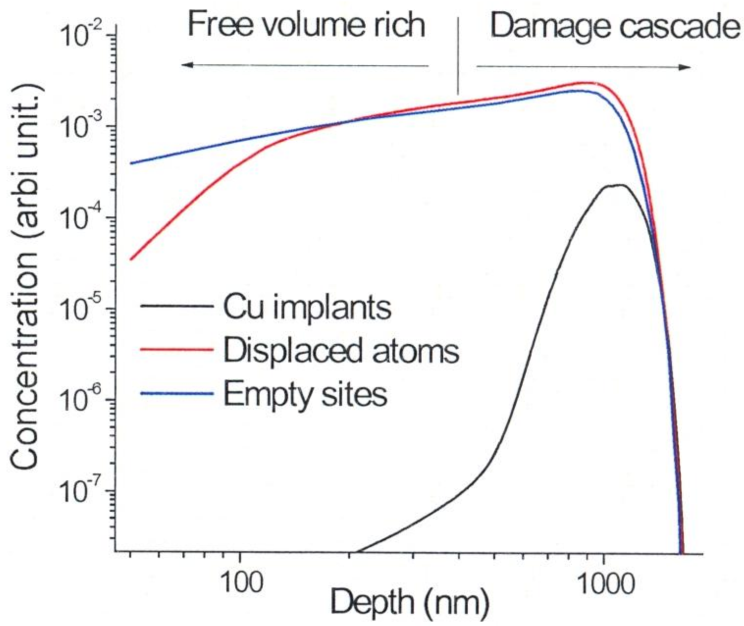

Figure 5: Depth profiles of Cu implants

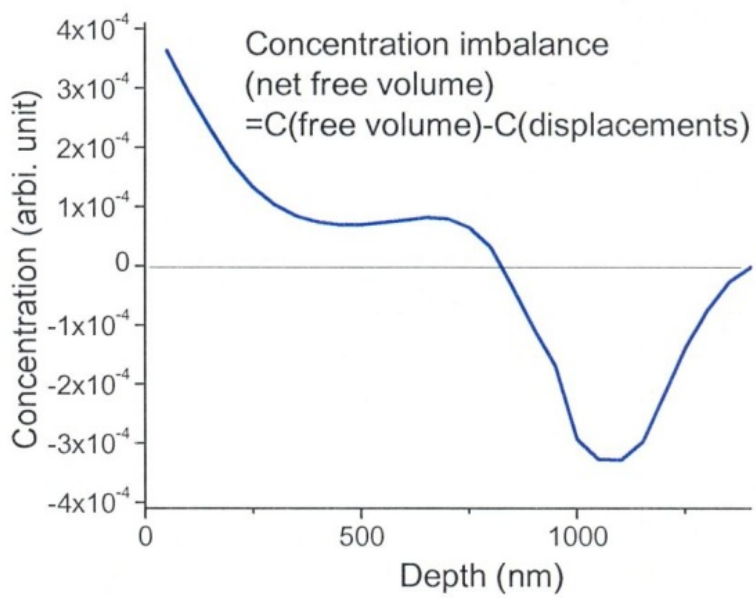

Figure 6: Concentration imbalance of free volume and displacement atoms

The experiment with Ion-irradiation at elevated temperature and cluster ion-irradiation were still under experiment. Those two experiments were worked by Mr. Balerio. I don’t have enough time to help him work on those two experiments.

CHAPTER IV

SUMMARY EXPERIMENT

As a result of the experiment, we can recognize the unique mechanical properties and deformation mechanism of MGs, and we are interested in their mechanical response. The relationship between atomic scale structure and their mechanical properties have not been understood clearly through extensive studies report [13]. Moreover, in order to investigate the mechanical response of amorphous metal, we have use large scale compression test [14], nano-indentation test [15], and micro & nano pillar compression test [16] to get a fully experiment about the mechanism properties of MGs.

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