

Fabrication of Rod Type $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ Bulk Amorphous Alloy by Warm Extrusion Process

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Cu-based bulk amorphous alloys have been considered as a new class of structural materials because of their excellent properties such as high strength, great elasticity and good corrosion and wear resistance. In addition, Cu-based bulk amorphous alloys have become more attractive for application as structural materials because of their relative low cost. Recently, bulk amorphous alloys with high glass forming ability were developed mainly in the Cu-, Ni-, Zr-, Ti-, and Mg-based alloy systems. However, due to the requirement of high cooling rate for the formation of amorphous phase from a liquid state, these alloys have not been used in the near net shape fabrication. The fabrication of bulk amorphous alloy by consolidation of amorphous powders provides an advantage of sample morphology because it enables the production of complex shapes without limitations on sample shape and dimension. Since significant viscous flow of the supercooled liquid makes it possible to consolidate the amorphous powders, amorphous alloy have a wide supercooled liquid region $\Delta T_x = T_x - T_g$ (where T_g is glass transition temperature and T_x is crystallization temperature). Many studies have been done on the consolidation of amorphous powders by various techniques including warm extrusion, hot pressing, equal channel angular extrusion, and warm rolling. In the present study, we report the fabrication of the $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ bulk amorphous alloys by warm extrusion of amorphous powders, prepared by a high-pressure inert gas atomization process.

To get bulk type amorphous alloy, $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ amorphous powders were produced by high pressure He gas atomization at a dynamic pressure of 4 MPa after heating to 1673 K using a guide tube with a hole diameter of 2 mm. The atomized powders were sieved using a conventional sieve analyzer and divided into the following five different size ranges: under 38, 38~45, 45~63, 63~90 and 90~150 μm . The $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ amorphous powders with a particle size below 63 μm were used for the fabrication of the bulk amorphous alloy. To conduct extrusion process, powders were filled in a copper can with an inner dimension of 20 \times 2 \times 50 mm in air, evacuated, sealed, and then pre-compacted in the warm extrusion system. Subsequently, the billet was heated with a heating rate of 50 K/min and holding time was about 5 minute. The extrusion temperature was 723 K and the extrusion ratio ($R=A_0/A$) was from $R=2$ to $R=5$. Structural characterization was performed by X-ray diffractometry (XRD). The thermal properties were examined by Differential Scanning Calorimetry (DSC). Test specimens with a dimension of 2 \times 2 \times 4 mm were prepared for compression tests. The compression test was conducted at room temperature and at a strain rate of $6 \times 10^{-5} \text{ s}^{-1}$.

DSC traces obtained from the atomized powders with below 63 μm size ranges during continuous heating at a heating rate of 0.67 K/s. The DSC traces exhibits glass transition temperature T_g of 712 K, onset temperature of first crystallization T_x of 764 K and integrated heat of crystallization of 66.5 J/g. The supercooled liquid region ΔT_x is about 52 K. Scanning Electron Microscope (SEM) micrograph of Cu-based amorphous powder with a size range of 30 ~ 63 μm . Most of the Cu-based amorphous powder shows a near spherical shape.

To determine an appropriate extruded condition, a time-temperature-transformation (T-T-T) curve was constructed with the $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ amorphous powders at different temperatures from 717 to 750 K. As can be seen in Fig. 3, with increasing annealing temperature, the onset time for crystallization decreases. The incubation time of maintaining the amorphous phase without crystallization at 723 K was 360s. In order to identify the amorphous forming ability of $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ bulk alloys with the ratio distribution, the XRD characterization was conducted.

Broad amorphous peak appears at the extrusion ratio range (excepting extrusion ratio 5) that indicate a successful formation of amorphous phases at the atomization condition. Variation in the hardness of $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ bulk alloy by warm extrusion according to extrusion ratio. It can be seen that as the hardness increases from 550 to 750 Mpa, the extrusion ratio increases from 2 to 5. TEM bright-field image and selected area diffraction (SAD). The TEM image of bulk alloy extruded at extrusion ratio 5 reveals the presence of nanocrystals of less than 20nm in size.

In the hardness result of extrusion ratio 5, sample out side is softer than the center region which means that out side of the sample was partially crystallized, subjected to more stress which in turn induced more heat exceeding T_x than the center region.

Stress versus strain curve of the bulk alloys tested under compressive condition at room temperature. The compressive load was applied in the transverse direction to the extrusion direction. The extruded bulk amorphous $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ alloy's compressive test showed a strength of 1.8GPa.

As a Result, $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ amorphous alloys were fabricated by warm extrusion of amorphous powders at the supercooled liquid region (723~743K) and extrusion ratio($R=A_0/A$) 2~5. The fully dense bulk alloys could be obtained by warm extrusion process, indicating that it is a successful consolidation process for Cu-based glassy alloys. Fully glassy $\text{Cu}_{54}\text{Ni}_6\text{Zr}_{22}\text{Ti}_{18}$ bulk alloys could be obtained at extrusion temperature of 723K with extrusion ratio of 2~5. Nano-scaled crystalline phase of less than 20nm was observed in the bulk alloys extruded at extrusion temperature of 723K with extrusion ratio of 5 by TEM image and X-ray diffraction pattern. The fracture strength for the fully amorphous bulk alloy was 1.8GPa and for the partial crystalline bulk alloy, 1.2GPa.

Keywords : amorphous, extrusion, bulk type