CHAPTER 5

MATERIALS AND METHODS

Materials

The materials used in the experiments consist of Tungsten and Copper powders from the METCO INC., Westbury, L.I., NY., and the standard specimens for microstructural analysis from AMS. The details of these materials will be presented in Appendix 1.

The equipment and conditions used in the experiments are

1. The equipment used in the Powder Metallurgy experiments consists of screening apparatus, hydraulic press, muffle furnace with temperature controller range from 500 to 1500 C , sintering atmosphere controlled apparatus, metallurgical microscope, etc.

2. The equipment used in neutron activation technique can be classified into 2 systems:

System I. Ge(Li) detector with 4098 channels ORTEC MCA model 8240 B.

System II. 3" X 3" NaI(Tl) solid type detector with 1024 channels CANNBERRA MCA model 8100 e.

3. The equipment used in X-rays Fluorescence Analysis can be classified into three systems:

- for EDX method,

  System III. Copper anode X-rays tube source with Xenon gas filled proportional detector and 1024 channels Tracor NORTHERN MCA model TN-1706.

  System IV. Isotopic annular X-ray sources with Si(Li) detector and 1024 channels NUCLEAR DATA MCA Series 2200.

- for WDX method,

  System V. Chromium anode X-ray tube source with Dispersive system using LiF (200 plane with 2d = 4.028 Angstrom) analysing crystal, thinned crystal NaI(Tl) detector and SCA.

4. The equipment used in density determination by gamma transmission technique consist of 10 mCi Cs-137 gamma ray source with 3" X 3" NaI(Tl) solid type detector and ORTEC SCA model 4890. The system of these equipment is illustrated as in Fig. 8.

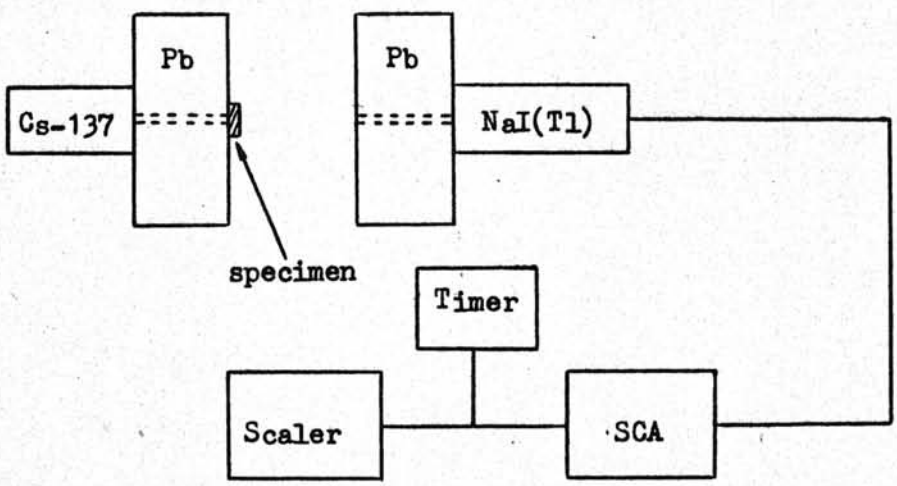

Fig. 8. The system used in density determination by gamma transmission.

Experimental Methods

These methods consist of:

1. Powder Metallurgy study and density determination by gamma transmission technique.

First of all, the size distribution of both powders (Tungsten and Copper) is determined by screen analysis, after that, a specific size of each powder (-38 microns for Tungsten powder and +53 to -75 microns for Copper powder) will be selected for further experiments. The selected powder of the two metals are mixed together at the ratio of 60:40 and 80:20 (Tungsten to Copper ratio) by using paraffin as a binder and benzene as a solvent. When the powders are already mixed and pressed in a die of 1/2 inch in diameter by a hydraulic press at the pressure about 8 tons per square centimeter and 12 tons per square centimeter, the green compacts (or pellets) are then sintered in a muffle furnace in a hydrogen atmosphere at the temperature of 1000 C and 1200 C for 1/2, 1, 1 1/2, and 2 hours, respectively, after heating to 800 C and soaked for one hour to allow all of the paraffin to evaporate. After sintering, the sintered compacts are slowly cooled in the furnace, then their densities are determined by gamma transmission technique, after that, these sintered compacts are ground, polished, and etched (using Murakami reagent) to study the microstructure changes during sintering.

2. Neutron Activation Analysis study.

The fixed weight of Tungsten and Copper powders are mixed together at a variation of compositions in polyethylene vials using a Mettler Analytical Balance model H54AR. After the vials are completely sealed, they are packed in a polyethylene rabbit and then activated in a thermal neutron flux of 10^{10} neutrons per square centimeter per second by using a pneumatic system. After activation is completed, the spectrum of the samples are analysed, especially, the peak of interested radionuclides by Detection System I or II. If the vials are packed in series and activated in thermal neutron flux of 5 Ci Pu-Be neutron source, they will be analysed by using the Detection System II. After analysing the results, a calibration curve of each condition will be constructed.

3. Method of X-ray Fluorescence Analysis:

- for EDX method,

First, a sample of sintered Tungsten-Copper pellet is used to determine the ability of each Detection System in separating the peak of Tungsten and Copper. If the resolution of the detector of any system is high enough to separate the energy peak of the two elements far apart, it will be used for analysing the chemical compositions. In the case of opposite results it will be rejected. The chemical compositions of the sample will be determined by using Covell's method as previously stated.

- for WDX method,

First of all, a sample will be scanned and a spectrum of the constituents showing relation between intensity and 2θ angles will be obtained. Due to its very high resolution compared with the EDX method, at least one peak in the presence of one element will be acquired. From this spectrum the most appropriate peak of each element will be selected. But in the condition of enterfering peak of other elements occured near the first required peak, another peak of lower intensity will be used in analysis instead of the main peak of interested elements. Manual Scanning will be taken to see the detail of the peak. By plotting a graph showing relation between intensity in a unit time interval and 2θ angles the apex of the photopeak will be known exactly. Then the 2θ angle at this point of a certain element will be fixed, and also the window of the SCA, and the LLD values will be plotted to determine the optimum condition of LLD value and the window size that will be used for that element all the time in the analysis. At this optimum condition, a calibration curve of intensity in a unit time interval and the concentration of that element will be constructed, and used as a standard curve by the help of a monitor. (In this experiment the standard specimens for microstructural study from AMS will be used in all of the WDX system experiment)