

CHAPTER 3

EXPERIMENTAL PROCEDURES

3.1 INTRODUCTION

This chapter describes the experimental procedure and equipment employed for sample preparation throughout this research project. This chapter comprises of the materials used and processes involved in sample preparations including the mechanical alloying process, consolidation techniques and heat treatment process under specific conditions. The latter section described both the specifications and capabilities of the laboratory equipment used and test procedures for analyzing the samples including XRD, EDX, SEM/FESEM, HRTEM, thermal analysis, densities and micro hardness measurement. These analyses are important in order to investigate the production factors to synthesized Ti-Al nano powders, the effect of MA process and heat treatment on the phase formation of Ti-Al intermetallics and its influence on the densities and mechanical properties of the alloys product.

3.1.1 Flowchart of Study

The flowchart of this research study are presented in figure 3.1. The process starts with mixing of Ti and Al elemental powder before milling in a ball milling equipment. Selected powder compound were analyzed by XRD, SEM/FESEM, EDX and HRTEM to observe the morphological changes and to identify phase and structural modifications. The powder then were consolidated into bulk form in order to measure the density and micro hardness alterations. This process were repeated again after heat treatment were completed. Thermal analyses were performed simultaneously during the heat treatment process.

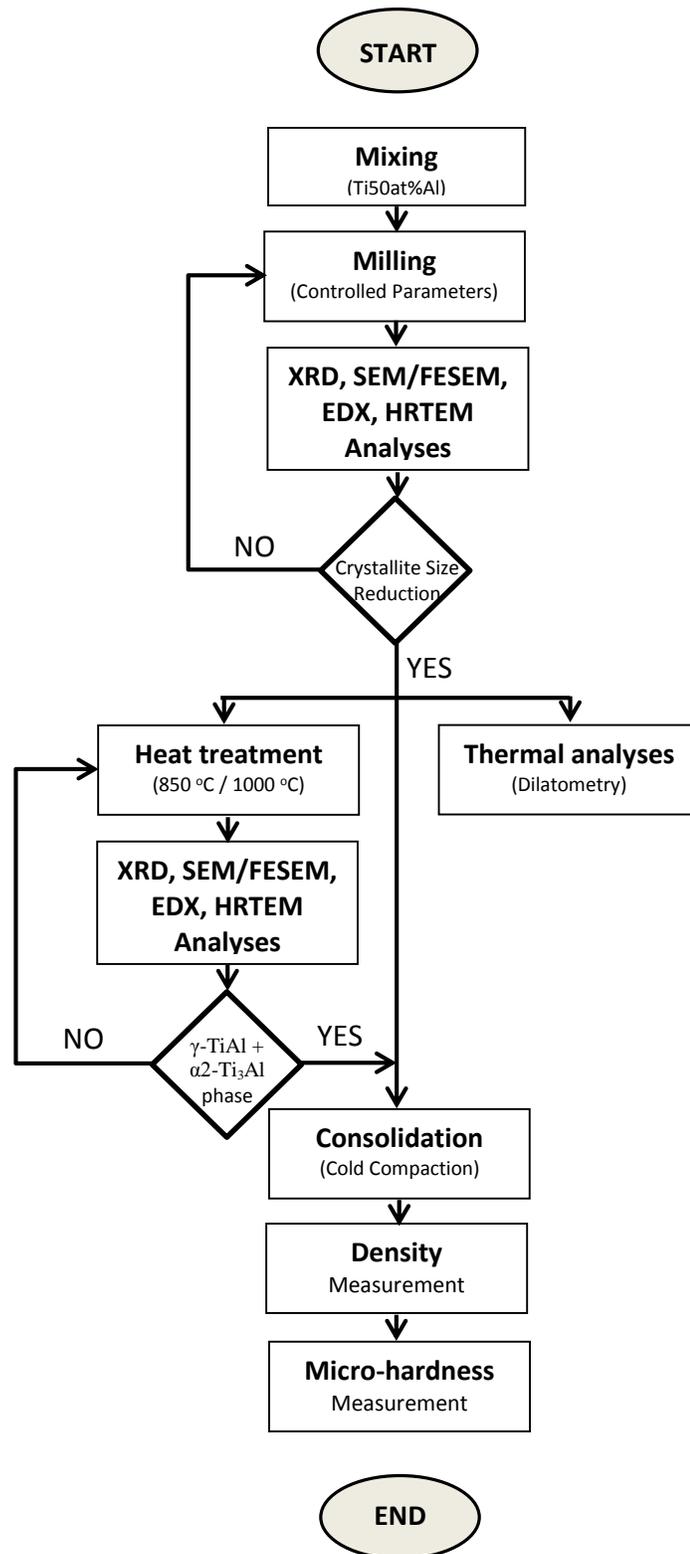


Figure 3.1: Research framework of this study.

3.2 SAMPLE PREPARATION

3.2.1 Materials

Commercial-grade elemental powders of 325 μm (100 mesh) Ti and 50 μm Al from Acros Organics, New Jersey, U.S.A. were used as starting materials with 99.5 % and 99.97 % of purity, and a molecular weight at 47.88 and 26.98 respectively (Figure 3.2). Since the primary focus of this study is to produce dual phase $\gamma\text{-TiAl} + \alpha_2\text{-Ti}_3\text{Al}$ phase which have more engineering significance, a composition of Ti50%Al of atomic percentage were use as the feed powder mixture. For each batch, the elemental powders were weighted in a ceramic vial on an electronic scale with an initial weight of 3.197 g for Ti powder and 1.803 g for Al powders to obtain a 5 g of powder mixture with Ti50%Al atomic percentages. The powder mixtures then were hand blended by using a spatula for a while to ensure the homogenous distribution between Ti and Al powder particles prior MA process.

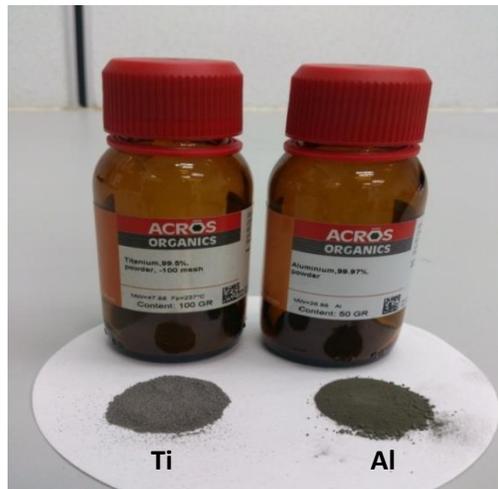


Figure 3.2: Elemental titanium and aluminum powder used as a feed materials.

3.2.2 Mechanical Alloying

The MA processes were carried out using a Retsch PM 100 planetary ball mill (Figure 3.3). One of the major issues associated with MA is the contamination of the powders from the milling tools. Therefore, throughout this research work, tungsten