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Fabrication and Analysis of Vanadium-Based Metal Powders for Selective Laser Melting

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Abstract

Vanadium Alloy is a type of advanced nuclear material with many ideal properties compared as traditional nuclear materials, which has very wide and important application in first-wall and blanket structural material for fusion power plant applications. So it has attracted increasing attentions, especially on new manufacturing methods, such as selective laser melting and so on. In this paper, the comparative study of the powders obtained by mechanical mixing method, dry grinding method and wet grinding method respectively was performed to evaluate the effect of ball milling process on the microstructure and degree of alloying of the vanadium-based powder mixtures with the nominal composition of V_5Cr_5Ti vanadium alloy. The powders prepared by dry grinding method exhibits better spherical-like morphology and degree of alloying than those prepared by mechanical mixing method and wet grinding method, which indicates that dry grinding method can be used to prepare the superfine vanadium alloy powders for selective laser melting. This work provides a new method as well as important insights into the preparation of superfine vanadium alloy powders for selective laser melting additive manufacturing technology.

Keywords

Vanadium-Based Metal Powders, Milling Process, Microstructure, Alloying Degree, Selective Laser Melting

1. Introduction

Vanadium Alloy has attracted increasing attention due to their wide range of applications in first-wall and blanket structural material for fusion power plant applications [1] [2] [3] [4]. Traditionally, vanadium alloy parts have been prepared by casting combined with forging method. However, there have been some technical bottlenecks in material preparation and parts molding, because vanadium alloys are extremely sensitive to oxygen, hydrogen and other impurities [5] [6] [7]. At the same time, some constraints such as long process flow, high comprehensive manufacturing costs and low material utilization also limited the preparation and application of vanadium alloys. So there have been urgent needs to develop new and green manufacturing technology with high quality and efficiency for manufacturing vanadium alloy parts.

Fortunately, the development of additive manufacturing (AM) provides a powerful tool to obtain vanadium alloy parts. This process is characterized by large temperature gradient and rapid cooling rate, which thus result in a significant no equilibrium solute-trapping effect that avoids component segregation and relieves solubility limitations. At present, AM has been used for the manufacturing of many alloy parts by means of pre-alloyed powder including Ti-6Al-4V, high speed steel and so on [8]-[12].

Selective Laser Melting (SLM) has emerged as one mainstream technology of AM, which possesses the technical capacity of directly manufacturing precise metal parts in the industrial field [13]. SLM Additive manufacturing technology provides a new thought and method for preparation and precise molding of vanadium alloy materials [14]. At the same time, SLM technology can greatly improve the material utilization and reduced the manufacturing cost of vanadium alloy parts. However, there is only coarse grain raw material or sheet of vanadium alloy at present market and no superfine vanadium alloy powder material with high quality applicable to SLM additive manufacturing [15] [16]. In this present work, the mechanical alloying method was employed to explore the preparation technology of superfine vanadium alloy powders with pure metal powders necessary for vanadium alloy material in order to meet the requirements of selective laser melting process. This work provides a necessary material basis for carrying out the investigation into SLM technique of vanadium alloys.

2. Experimental Procedures

Elemental powders with the average diameter of 35 μm including pure vanadium powder (purity ≥ 99.98%, granularity < 425 mesh), pure chromium powder (purity ≥ 99.98%, granularity < 425 mesh) and pure titanium powder (purity ≥ 99.9%, < 425 mesh) were accurately weighed to get the desired compositions V_5Cr_5Ti. The alloy was prepared through three different methods, which were mechanical mixing method, dry grinding method and wet grinding method respectively. Pre-weighed powder mixtures were canned into a QM-1SP4-CL high energy planetary ball mill machine by mechanical mixing method. Then, a high-energy ball milling process was performed at the rotation speed of 350 rpm for 1 h and this process used stainless-steel balls with a ball to powder weight ratio of 1.5:1, and the prepared powder sample was labelled as PM. Pre-alloyed Vanadium alloy powders were firstly prepared by dry grinding method. The high-energy ball milling process was performed at the rotation speed of 350 rpm for 5 hours, 10 hours, 15 hours, 20 hours and 25 hours, and the prepared powder samples were marked as D05, D10, D15, D20 and D25, respectively. This process used stainless-steel balls with a ball to powder weight ratio of 10:1. Secondly, another type of pre-alloyed vanadium alloy powders was prepared by wet grinding method. The high-energy ball milling process was performed at the rotation speed of 350 rpm for 5 hours, 10 hours, 15 hours and 20 hours, and the prepared powder samples were marked as W05, W10, W15 and W20, respectively. This process used stainless-steel balls with a ball to powder weight ratio of 10:1 and Acetone was added as a process control agent (PCA) to prevent excessive cold welding amongst the powder particles.

The property of the as-prepared powder samples was examined by X-ray diffractometry (XRD, D/max-RB, Ri-gaku, Japan) with Cu-Kα radiation. Scanning electron microscopy (SEM, LEO1530) with EDS analysis was used to examine the morphology of powders and the compositions of the samples. The particle size of all powder samples were examined by Laser diffraction particle size analyzer (Mastersizer 2000, Malvern instruments company, England).

3. Result and Discuss

The SEM images of vanadium-based powder mixtures with the nominal composition of V_5Cr_5Ti by mechanical mixing method and pre-alloyed vanadium alloy powders with the nominal composition of V_5Cr_5Ti by dry grinding method with different time were shown in **Figure 1**. It can be seen that there are no obvious change of the mixed powder particle size and its morphology for PM sample, which suggest there should be low energy for mechanical mixing method and the impact of ball on powders maybe is less than enough to make the metal powder deformation.

By comparing the SEM morphology of the mixed powders marked as PM with the pre-alloyed vanadium alloy powders including D05, D10, D15, D20 and D25, the change of the powder particle size and morphology of the powders can be easily observed. After dry grinding for 5h, the powders are consisted of large flat particles and small flat particles as displayed in Fig 1, *i.e.* D05 powder sample. It can be seen that the average particle size of powders after dry grinding for 10h is smaller than those of the D05 powders by dry grinding method and the particles change into spherical-like morphology. With the increase of dry grinding time (D15, D20, D25), the morphology of powders by dry grinding method get much more spherical. On the other hand, the change of particle size can be clearly observed, *i.e.* the particle size quickly gets small with the increasing of dry grinding time from 15 hours to 25 hours. According to the above results, the morphology evolution of the pre-alloyed vanadium alloy powder by dry grinding method is obvious with the increase of grinding time. Besides, when the dry grinding time is 5 hours, D05 powder sample has the particle size distribution with double peak.

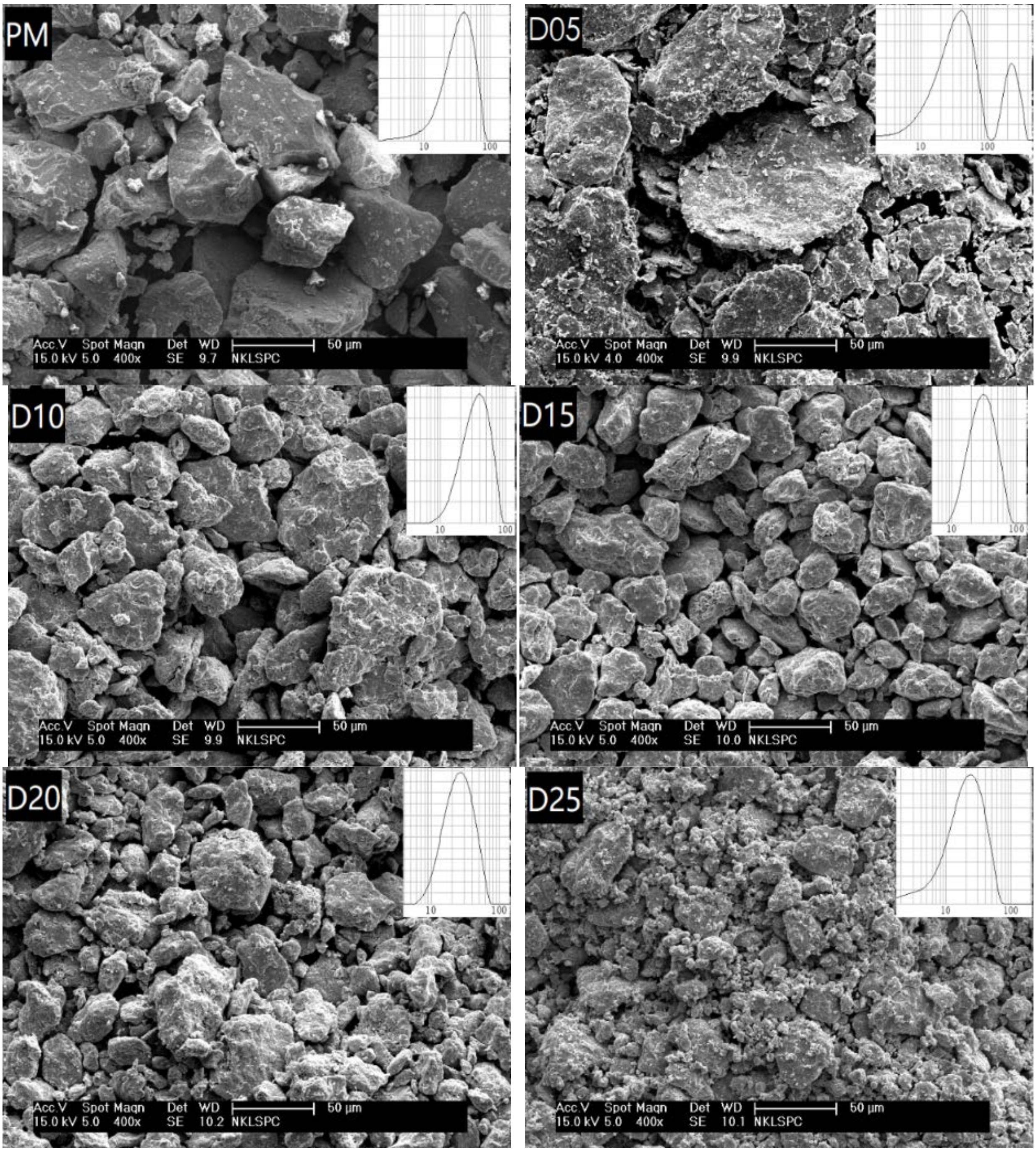

Figure 1. Morphology and particle size distribution of vanadium-based powder mixture with the nominal composition of V_5Cr_5Ti by mechanical mixing method and pre-alloyed vanadium alloy powders with the nominal composition of V_5Cr_5Ti by dry grinding method with different time (5 hours, 10 hours, 15 hours, 20 hours and 25 hours). Note: the particle size distribution of prepared powders is inserted at the right and upper corner of every SEM image.

For a further investigation into the particle size change of vanadium alloy powders by dry grinding method, laser diffraction particle size analyzer (LDPSA) has been used to study the particle size for different dry grinding time in terms of their average diameter. According to the LDPSA results (**Figure 2**), it can be found that the change of particle size for dry grinded powders showed a

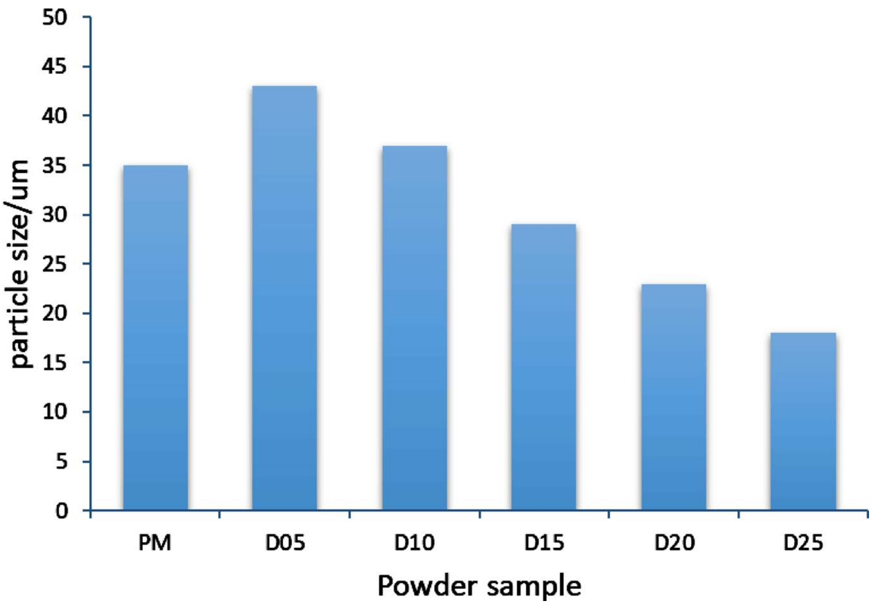

Figure 2. The particle size of prepared vanadium alloy powders with different dry grinding time.

tendency to increase first and then decrease. However, there is no obvious change of the particle size and morphology for mechanical mixed powders, *i.e.* PM sample, compared with initial elemental metal powders. So the LDPSA results are consistent with the morphology of powders by SEM.

The morphology and particle size distribution of pre-alloyed vanadium alloy powders with the nominal composition of V_5Cr_5Ti by wet grinding method with different time were shown in Figure 3. Figure 4 shows the particle size of pre-alloyed vanadium alloy powders with different wet grinding time. For W05 powder sample, the particle surface is quite flat, and the different particles are difficult to be distinguished. It can be seen from Figure 3 that the particle size of vanadium alloy powder shows a tendency to decline first and then increase with the increasing of grinding time within 10 hours, but decreases rapidly with further increase of grinding time beyond 10 hours, according to W10, W15 and W20 samples. It was noticed that compared with dry grinded vanadium alloy powders, the pre-alloyed powders by wet grinding have the shape of thin plate, and the particle size is much smaller than powders of PM powder samples when grinding time is more than 15 hours, *i.e.* W15 and W20 powder samples, as shown in Figure 3 and Figure 4. Besides, when the wet grinding time increases to 10 hours, W10 powder sample has the particle size distribution with double peak. By comparing Figures 1-4, it can be found that whether dry grinding powder or wet grinding powder, its particle size is maximal as long as the particle size distribution has double peak.

Figure 5 shows the XRD spectra of the dry grinded powders and mechanical mixed powders. It can be found that sharp diffraction peaks are clearly observed in mechanical mixed powders, which are identified as Cr, Ti and V, respectively. However, the relative intensity of the peaks corresponding to Cr phase keeps

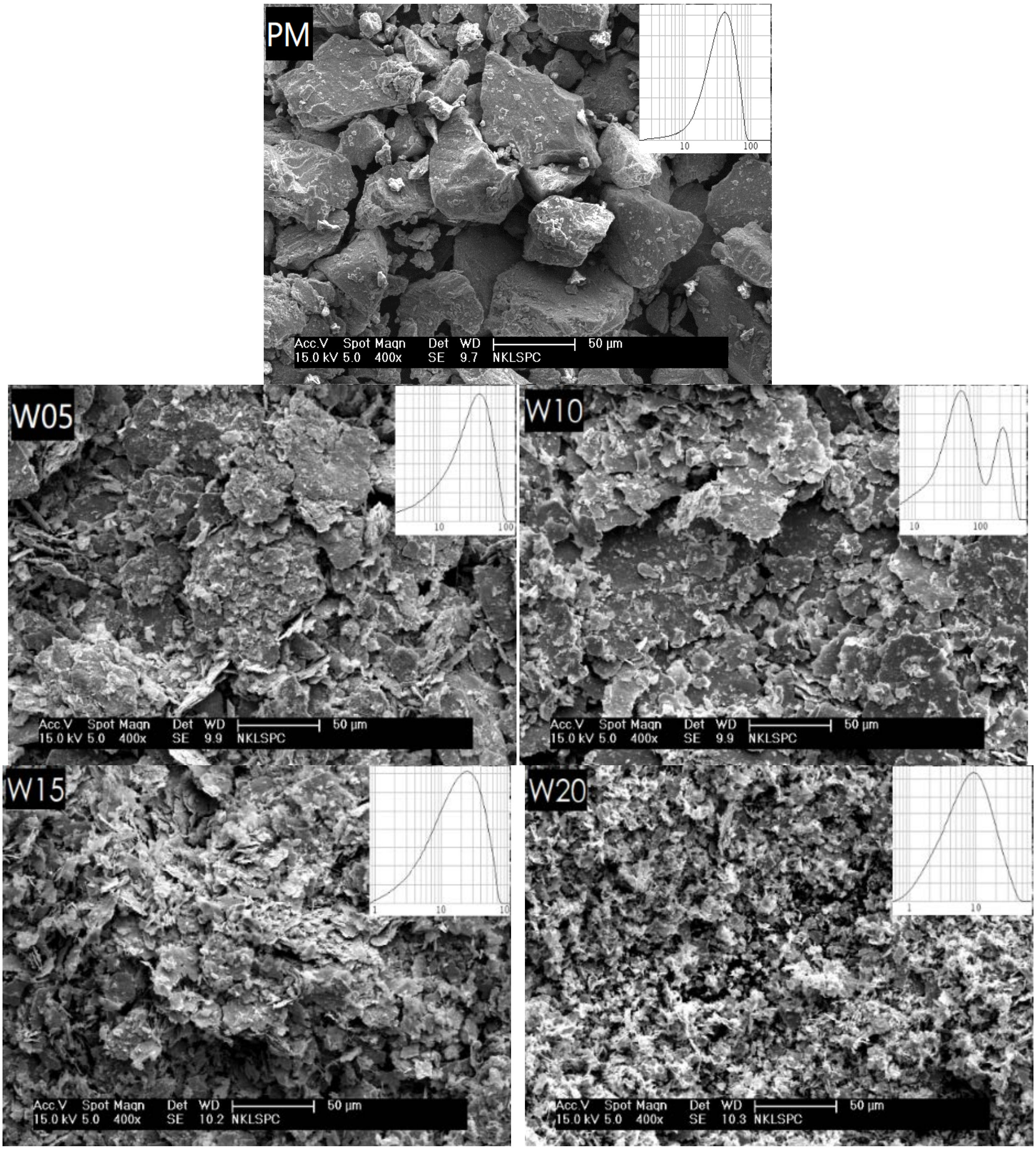

Figure 3. Morphology and particle size distribution of pre-alloyed vanadium alloy powders with the nominal composition of V_5Cr_5Ti by wet grinding method with different time (5 hours, 10 hours, 15 hours and 20 hours). Note: the particle size distribution of prepared powders is inserted at the right and upper corner of every SEM image.

decreasing with the increasing of dry grinding time. In addition, the peaks corresponding to Ti phase disappear when the dry grinding time reaches 5 h, which means that during the dry grinding process Ti phase and Cr phase have started to be solid soluted in V phase. Besides, when the dry grinding time exceeds 10 h, some new peaks appear. After calibration, these emerging peaks were identified to be corresponding to V (Cr, Ti) phase, which indicates the partial alloying of

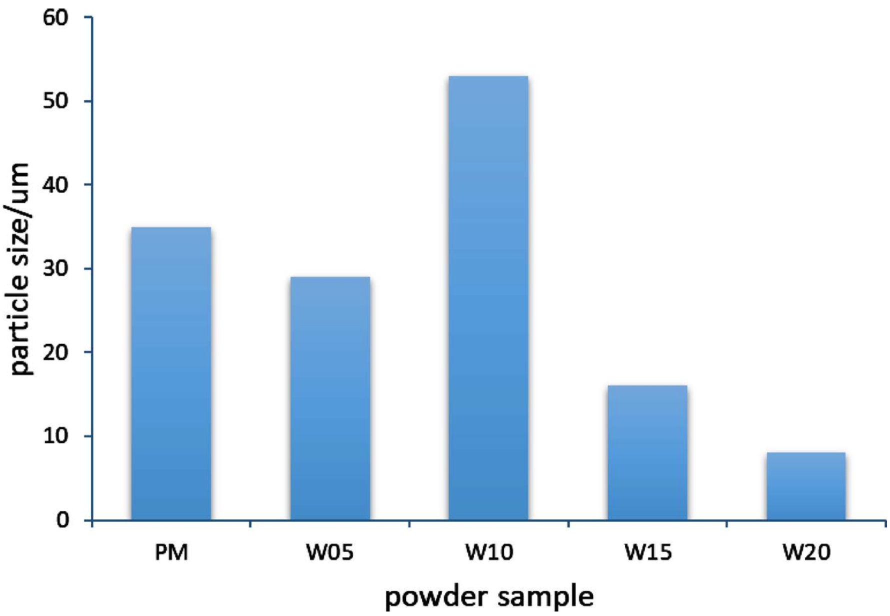

Figure 4. The particle size of prepared vanadium alloy powders with different wet grinding time.

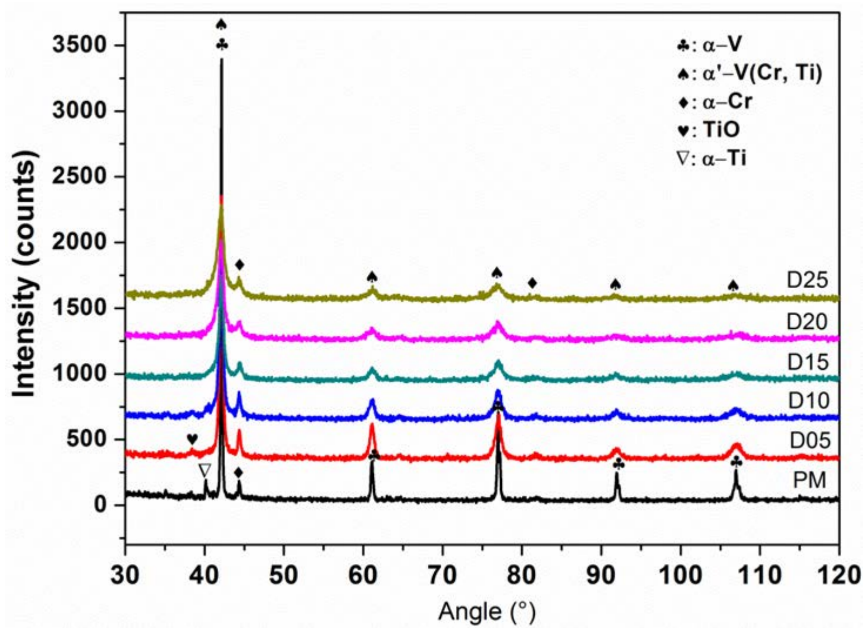

Figure 5. The spectra of mechanical mixed and dry grinded powders.

V. According to the XRD results, it can be found that the pre-alloyed vanadium alloy powders by dry grinding method is constituted by V (Cr, Ti) phase, V phase and Cr phases.

Figure 6 shows the XRD spectra of the wet grinded powders and mechanical mixed powders. By comparing it with the XRD spectra of dry grinded powders, the relative intensity of the peaks corresponding to Cr phase still strong for powders with wet grinding time of 20 hours. According to the XRD results, it can be found that the pre-alloyed powders by wet grinding method have a lower

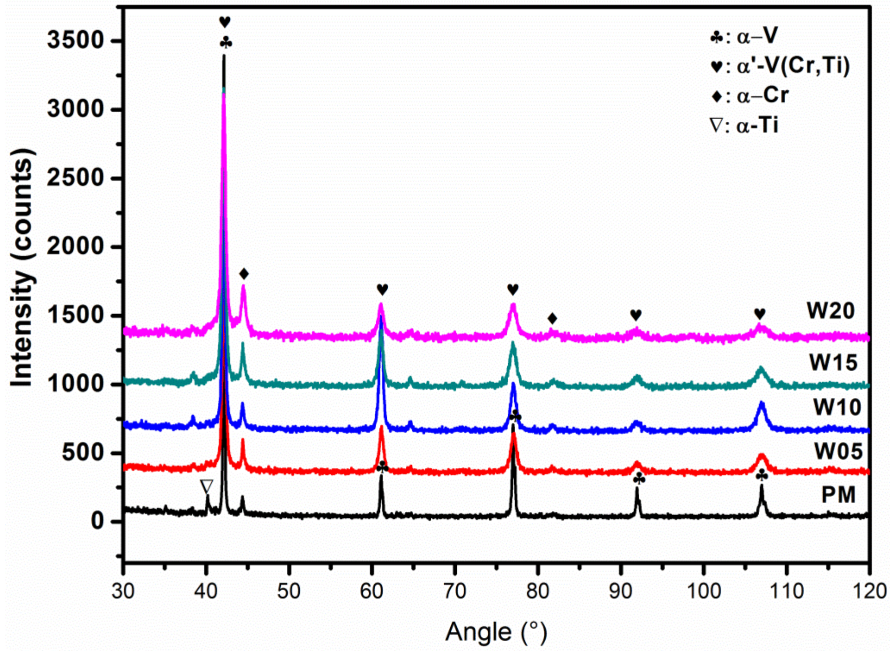

Figure 6. The spectra of mechanical mixed and wet grinded powders.

degree of alloying than dry grinded powders due to reducing the elements diffusion rate by Acetone cooling effect.

4. Conclusion

The comparative investigation of the powders obtained by mechanical mixing method, dry grinding method and wet grinding method, respectively, was performed to evaluate the effect of ball milling process on the property of vanadium-based powder mixtures with the nominal composition of V_5Cr_5Ti vanadium alloy. It is shown that the pre-alloyed V_5Cr_5Ti powders by dry grinding method have a higher degree of alloying than wet grinded powders and mechanical mixed powders. Furthermore, dry grinding method can efficiently decrease the granularity of vanadium-based powder mixtures with the nominal composition of V_5Cr_5Ti vanadium alloy that is expected to be contributive to selective laser melting. After dry grinding 15 hours, the particles change into spherical-like morphology. With the increase of grinding time to 25 hours, the morphology of powders by dry grinding method gets much more spherical, and the granularity of particles decrease sharply. This work provides a new method as well as important insights into the preparation of superfine vanadium pre-alloyed powders for selective laser melting.

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