

Recycling Process of WC Fine Powder Contained by Cemented Carbides Parts in JAPAN

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Abstract

Cemented carbides material (WC-Co hard alloy) were recognized very important and expensive tool or die assembly parts because of compose for the main elements of rare metal (W and Co etc). This research was developed to separate and recover of WC fine powder contained by WC-Co materials. Recycling process was a new method named by the Tin impregnation for decobaltification on cemented carbides. This reaction occurred to product a brittle Co-Sn intermetallic compounds, thereafter it carried out by acid cleaning solution and physical milling or powdering. New process was able to recover about 60% WC fine powder from 1 to 5 μ m.

1. Introduction

Cemented carbides material are mainly utilized in cutting tool and die material in Japan, and are products of powder metallurgy. This material were composed by WC, and added to use Co element for binder. The characteristics of this material have a high hardness number (HRA 80~93), excellent wear and corrosion resistance. On the other hand, WC-Co alloy were a very expensive parts from 25,000~40,000 yen/kg in Japan because of compose for the main elements of rare metal (W and Co). Therefore, recycling technique of this alloy was very important, but this technique was nothing at present time because the physical properties is a excellent value and it's crushing process is very expensive. Then, we considered a very useful process to recycle the used cemented carbides.

This research was a new recycling process by utilized in Tin impregnation method. The characteristics of this process were first product a brittle Co-Sn intermetallic compounds, next this compounds carried out by repeat to cleaning by the acid solution and physical milling or powdering. This research investigate to recover as possible as WC fine powder contained by cemented carbides.

2. Experimental procedure

Cemented carbides material on this experiment was composed WC and 13mass% Co. The grain size of WC was from 1 to 4 μ m, hardness number (HRA) was 87.3. Metal bath was used to pure Sn particle and melted the fixed temperature. The sample were impregnate by melted Sn bath. This reaction occurred to product Co_3Sn_2 (γ' phase) intermetallic compounds by react between Sn and Co. As a results, the decobaltification of WC-Co alloy occurred from the surface. Reacted sample were repeat to crushing and stirring by 18vol% HCl solution. It's treatment were dissolved by γ' phase and melted Sn during HCl solution. Recycled WC fine powder were measured the recovery ratio, powder size and impurity element by a qualitative analysis of EPMA method.

Finally, it was investigated and compared the characteristics between the recycled WC powder and commercial WC powder.

3. Experimental results

Fig.1 shows the cross-section sketch and SEMmicrographs of 3 types treated temperature after Sn impregnation. After Sn impregnation, it consist two area to separate WC-Sn layer near reaction interface and non-reaction WC-Co base alloy in center part. WC-Sn layer was become thicker by raise the treated temperature. From the reduction in cross-section area was non-reduced, this process indicate to the non-reduction of

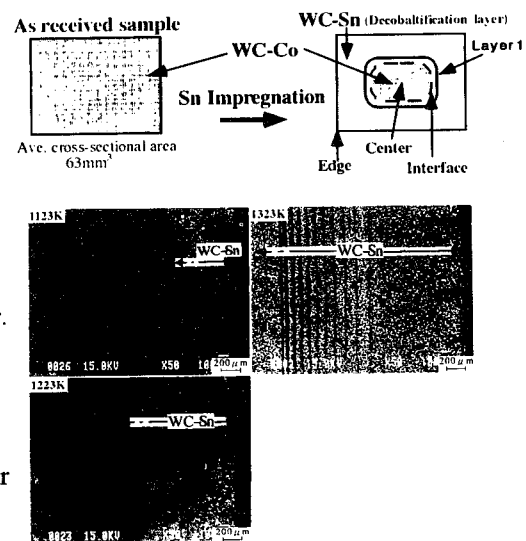


Fig.1 Cross-section sketch and SEM micrograph of 3 types treated temp. by Sn impregnation

particle except to Co element. On the other hand, WC-Sn layer indicate to lamellar reduction from surface, it identified as γ' phase (Co_3Sn_2) appeared black layer, other parts (non-black part) changed from Co to Sn. These layer identified to WC particle and the surplus Sn alloys.

Fig.2 shows the relation between decobaltification layer and holding time at 1323K constant by the Sn impregnation method. Decobaltification layer shows to relate as the second power of the holding time by the diffusion's rule. From this results, the measurement of minimum sample size were required to removing time of Co element from cemented carbides. This WC-Sn alloy was able to easy crushing and powdering both surplus Sn and γ' phase by automatic milling machine. As a result, this alloy is classified under $45\ \mu\text{m}$ particles. On the other hand, the surplus Sn and γ' phase contained by crushed particle was done to stirring and cleaning by 18vol% HCl solution, dissolved Sn and Co (γ' phase) was able to recover more WC fine powder only. The powder over $45\ \mu\text{m}$ was repeated to crushing, stirring and cleaning by acid solution, the recovery ratio of WC fine powder is raised as possible.

Fig.3 shows the relation between recovery ratio of WC powder and treated temperature. In this experiment, it obtained to 55% recovery ratio at 1323K-7.2ks. These WC powder show the grain size from 1 to $5\ \mu\text{m}$, and didn't analyzed to retained Sn and Co used by Sn impregnation method. It was able to recover almost perfect WC fine powder. But, the surface of recycled WC powder by this treatment observed to consist to some oxygens. Therefore, physical properties was reduced compared to the commercial WC powder. When this recycled powder use as raw powder, it is necessary to treat hydrogen reduction treatment.

Fig.4 shows the flow chart of this recycling process on cemented carbides. First, the purpose of decobaltification, molten Sn impregnation was treated at 1323K. Second, obtained WC-Sn alloy was repeated to physical milling and stirring+ cleaning used by 18vol% HCl solution when powder size made under $45\ \mu\text{m}$. As a result, recycled WC powder get to recovery over 60%. But, the surface of WC powder consist to some oxygen. It is necessary to treat the hydrogen reduction ($1073\text{K}-3.6\text{ks}$). Then, recycled WC powder added to C and Co element as same as the commercial alloys, thereafter treat to mixing among 3 element, compaction and sintering. It is able to recycling by 8 step process and to gain good physical properties of cemented carbides.

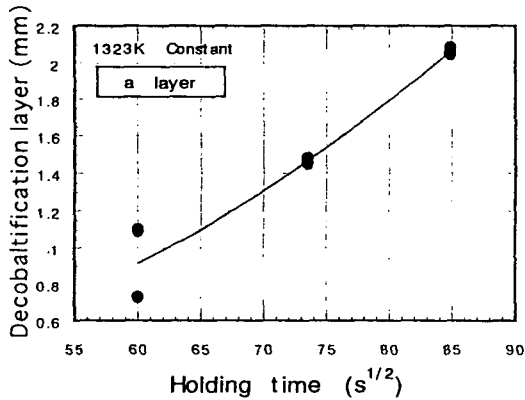


Fig.2 Relation between decobaltification layer and holding time at 1323K constant.

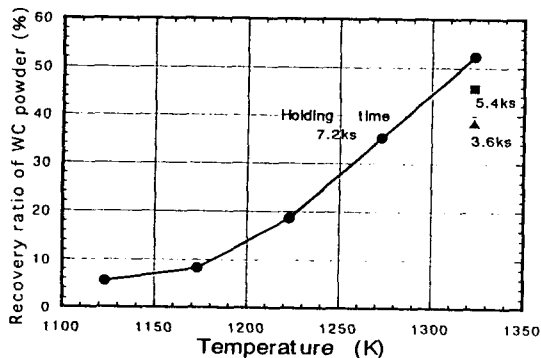


Fig.3 Relation between recovery ratio of WC powder and treated temperature.

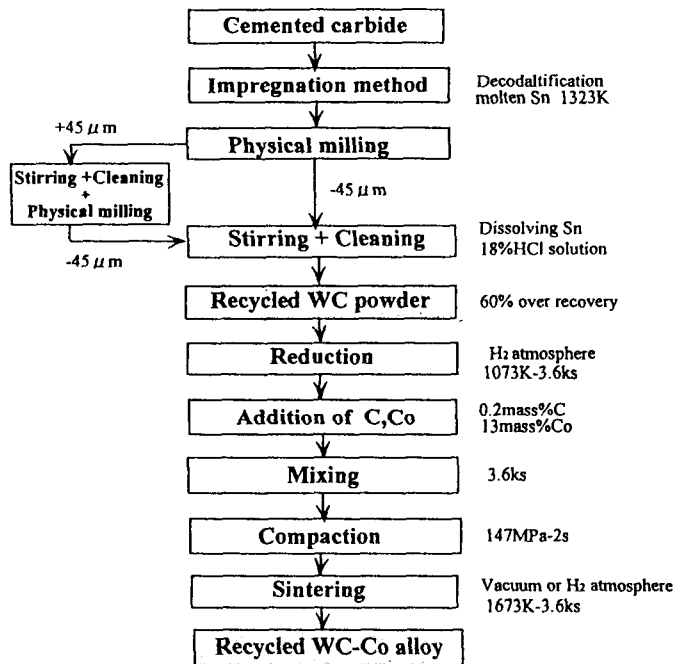


Fig.4 Flow chart of recycling process on Cemented carbides.