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XPS and EDS Analysis of VC Coatings Prepared by TD Process

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Abstract: VC (vanadium carbide) coating was prepared on the substrate surface of Cr12MoV cold worked die steel by TD (thermal diffusion) process using anhydrous borate, FeV50, FeSi45 as main materials. The structures and the chemical element distributions of the coating were characterized with SEM (scanning electron microscope), XRD (X-ray diffraction), XPS (X-ray photoelectron spectroscopy) and EDS (energy dispersive spectrometer), respectively. The chemical element distributions of VC coating surface and interface were analyzed with plane scan of EDS, and the mechanism of interfacial metallurgical bonding was discussed. The results show that the VC coating is a compound, consisting of C and V elements, and the concentration of V atom is twice as large as that of C atom. The VC coating is single VC phase, where the electron binding energy of V element is located at 512.8 eV, and that of C element is located at 282.2 eV. The diffusion between V, C and Fe elements occur at the coating interface and a layer of interface diffusion is formed, and the interface of VC coating-substrate is combined with the metallurgical bonding.

Key words: VC coating; TD (thermal diffusion) process; bonding interface

VC coating is one of the highest hardness transition metal carbon compounds and also a stoichiometrical compound ^[1,2]. It has a face-centred cubic structure with high melting point, high hardness and high chemical stability, etc.^[3] Therefore it is widely used in materials modification. There are many methods of preparing VC coating, such as direct carbonization, in situ burning form, and TD (thermal diffusion) processing^[2-4]. VC coating can be formed in the mold surface by TD process, which is one of surface treatment methods of solving surface pulled damage of forming die^[4]. At present, TD surface strengthening process is widely used for moulds in Japan, Korea and America. However, the researches on TD process are still at experimental stage, and used rarely in the actual production, which somewhat restrict the application of TD process in industries. The investigations about TD process

mainly focused on growth mechanism, structure, abrasion performance of VC coating^[5-8]. The related chemical element distributions in VC coating showed that C concentration had a slight increase in coating interface, while C content first slightly increased to the maximum capacity, presenting a decreasing trend ^[9]. But there was little report about the chemical element plane distributions of VC coating interface and metallurgical bonding formed by interface diffusion. In the present investigation the VC coating was prepared on the Cr12MoV cold worked die surface by TD process. The structures and the chemical element distributions were analyzed with SEM, EDS, XRD and XPS, respectively, and the metallurgical bonding mechanism of the coating interface was analyzed to provide the basic experiments for actual applications.

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1 Experiment

The substrate material was Cr12MoV steel, and the equipment was a TD salt bath furnace with the highest heating temperature of 1300 °C and automatic control temperature accuracy of \pm 5 °C. The raw materials were composed of the following powders: (1) no-water borax salt, the particle morphology is shown in Fig.1a, and the average size was 20~30 µm; (2) reducing agent FeSi45, its particle morphology is shown in Fig.1b, and the average size was 10~15 µm; (3) vanadium agent FeV50, its particle morphology is shown in Fig.1c, and the average size was 20~25 µm. The XRD patterns of raw materials are shown in Fig.2.

When the salt bath was prepared, first of all, the salt furnace was started by base salt. Then the vanadium agent of FeV50 and reducing agent of FeSi45 were mixed evenly into salt bath, and finally the activator of NaF was put in to increase the liquidity of salt bath. After full melting, Cr12MoV cold worked die steel was soaked in salt bath at the temperature of 1200 °C for 12 h, afterward quenched in oil at the tempering of 180 °C for 2 h, and then the required sample was obtained. The surface-interface morphologies and chemical element distributions were observed and tested with JSM-6360 LA typed SEM and the electronic energy equipment. The phase of VC coating and the XPS map were analyzed by D/max2500PC type XRD and ESCALA 250 type XPS, respectively.

2 Analysis of Experimental Results

2.1 Surface morphology, EDS and XRD analysis

The coating surface is smooth and dense, as shown in Fig.3a. The distribution of grain and particle size is uniform with certain porosity. EDS analysis of the surface is shown in Fig.3b. The coating is composed of V and C elements without other impurities, and the mass fraction is V=89.45%, C=10.55%, and the atomic fraction is V=66.66%, C=33.34%, which is the main reasons for that VC coating has a high hardness. The microhardnesses of coating surface and substrate surface are 30500~32000 MPa and 5000~5500 MPa under the loading of 2 N for 15 s, respectively, measured by HXD-1000 type micro hardness tester, forming the function hardness gradient that improved effectively the surface resistance strain capacity and the surface quality of forming die.

The phase structure was analyzed with D/max2500PC type X-ray diffraction, the test parameters were: Cu target K α radiation (λ =0.15418 nm), pipe voltage of 40 kV, current of 100 mA. The results are shown in Fig.4, and there is single VC phase with a little amount of V₈C₇ and C atoms, no brittle α -FeV phase, which is the same result as



Fig.1 Surface morphologies of the salt raw materials: (a) borax, (b) silicon iron powder, and (c) vanadium iron powder



Fig.2 XRD patterns of the raw materials: (a) borax, (b) silicon iron powder, and (c) vanadium iron powder

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