Tribological Evaluation of Die Materials for High Velocity Compaction of Stainless Steel and Stellite Powders

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Abstract It is known that gas atomized powders as well as highly alloyed metal powders are difficult to press into green bodies using conventional quasi static pressing techniques. However, the development of the high velocity compaction (HVC) technique has expanded the possibility to compact a larger spectrum of metal powders to PM components of high density. As with conventional pressing techniques, transfer of powder material to the die walls is a common problem in HVC. This is especially true when pressing metal powders with no or a very low content of internal lubricant as well as highly alloyed metal powders which usually have a strong tendency to adhere on the die walls of the tooling. As a result, the characterisation of the material transfer tendency of different types of metal powders to potential die materials is of outmost importance. In the present study the material transfer tendency between austenitic stainless steel and Stellite powder and some potential die materials including two different high speed steels and four different PVD coatings have been evaluated. The test method is based on controlled scratch testing using a commercial scratch tester where the powder particles are drawn over the surface in a well controlled sliding contact. The results show that of the materials evaluated a low friction WC/C coating shows the best performance and is able to avoid material transfer also when unlubricated powders are used. In contrast, conventional nitride based PVD coatings such as TiN, (Ti,Al)N and CrN show no positive effect when it comes to reducing the material transfer tendency. Of the HSS grades evaluated, a nitrogen alloyed ASP 2040 shows the best result.

1 INTRODUCTION

The mechanical properties, and especially the fatigue properties, of powder metallurgy (PM) components are significantly improved by reducing the porosity. As a result, new powder compaction methods such as high velocity compaction (HVC) have been developed in order to obtain PM components of higher density. In the HVC process densification is achieved by intensive shock waves, created by e.g. a hydraulically operated hammer, that transfer the compaction energy through the compaction tool to the powder. The mass of the hammer and its velocity at the moment of impact determine the compaction energy and the amount of densification and its has been found that green densities above 7,6 g/cm^3 can be obtained for Astaloy Mo +2% +0,6%C [2] and above 7,2 g/cm^3 can be obtained for stainless steel 316 [1]. A natural step to further increase the green density and simplify the sintering process is to reduce the amount of internal lubricant in the powder. However, this will significantly increase the adhesive contact between the punch/die and the powder/green body during the powder compaction process and as a result material transfer from the softer metal powder to the harder tool surfaces, causing galling, may occur. In the HVC process this problem is most severe on the die walls and increases with increasing green densities. As a result, the compaction and ejection forces as well as the wear rate of the die and punch surfaces will increase. Besides, the quality of the green compacts may be reduced due to an increased defect density. Thus, tool materials optimized for high velocity powder compaction processes must fulfil a number of requirements.

Due to the heavy shock loads during HVC, a high toughness of the tool material is of high importance. As a result, PM tool steels are often used in HVC because its higher toughness compared to e.g. cemented carbides. However, a high toughness is usually accompanied by a lower hardness that in turn may lower the wear resistance, especially in the compaction of high alloyed metal powders. This problem can be solved by the use of thin hard coatings deposited on a tough substrate material. Unfortunately, a hard and wear resistant coating does not necessarily solve the problem with material pick-up and galling, it also needs to show a low affinity to the metal powder material, i.e. intrinsic low friction properties [3, 4, 5, 6, 7]. The selection of a suitable coating is further limited by the tool material used and the tool design. CVD coatings are usually deposited at a high temperature, typically 900-1100 °C, exceeding the tempering temperature of the tool steel requiring post-hardening of the tool. PVD coatings, on the other hand, are deposited at significantly lower temperatures, typically 200-450 °C, i.e. below the tempering temperature of the tool steel. However, a drawback of the PVD process is its line-of-sight principle, i.e. the surface to be coated need to be faced from the target, which means that PVD is less suitable for coating of die cavities that has a high height/diameter ratio. As a result, the interest for newly developed low friction tool steel grades have increased during the last years [8].

Manufacturing of tooling for testing of different die material is a time consuming and expensive route to take. Instead several different laboratory testing techniques for characterizing the tribological properties of different tool materials in a controlled environment are frequently used, e.g. button-on-block, button-on-cylinder [9], pin-on-disc, cylinder-on-cylinder and load-scanner [10]. In the present study, a new approach for evaluating the tribological conditions in powder compaction has been used in order to evaluate the friction characteristics and material transfer tendency between metal powders and potential die materials, including different HSS grades and PVD coatings. The method is based on controlled scratch testing using a commercial scratch tester but instead of the commonly used Rockwell C diamond stylus powder particles are drawn over the surface in a well controlled sliding contact.

2 EXPERIMENTAL

2.1 Materials

Two different powder metallurgy high speed steel (HSS) grades, ASP 2040 and ASP 2053 (Erasteel Kloster AB designation) and four different PVD coatings were evaluated in the present study, see Table I. While the ASP 2040 is a nitrogen-alloyed low friction HSS steel grade the ASP 2053 is a conventional HSS steel grade. The ASP 2040 grade was heat treated by austenitization at 1100 °C followed by tempering two times for 1 h at 560 °C while the ASP 2053 grade was heat treated by austenitization at 1180 °C followed by tempering three times for 1 h at 560 °C. The resulting microstructures consist of a martensitic matrix with fine (1 µm in size) carbides/carbonitrides and carbides, respectively, see Fig 1.

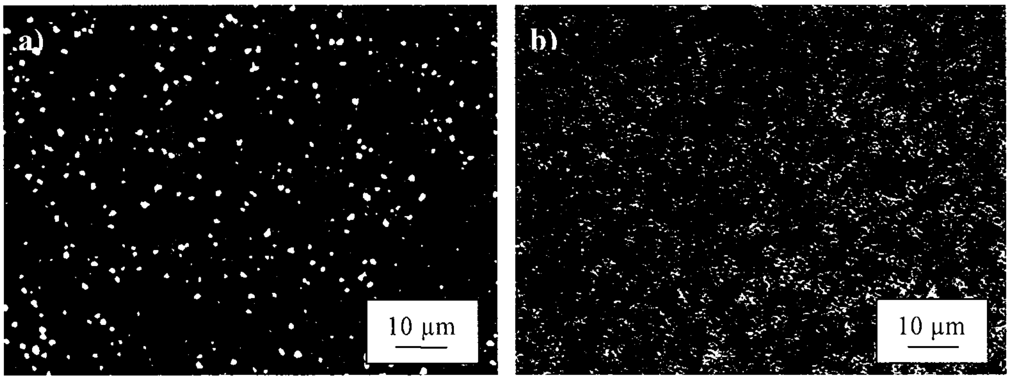

Figure 1. Microstructure of the HSS grades investigated as observed in the SEM (backscatter electron images). a) ASP 2040 showing black MN/MC-phase and white M_6C-phase in a martensitic matrix. b) ASP 2053 showing mainly black MC-phase in a martensitic matrix.

The different PVD-coatings, represent three traditional hard metal nitride coatings and a low-friction carbon rich metal carbide coating commonly used on different types of forming tools. The PVD coatings were deposited by Balzers Sandvik Coating AB, Sweden, using high plasma density activated reactive ion plating (metal nitride coatings) and reactive dc magnetron sputtering (metal carbide coating), respectively. While the former coatings were deposited as single layer coatings the latter coating was deposited as a multilayered structure of WC and C. In order to promote the adhesion of the latter coating to the substrate a Cr boundary layer was first deposited. All PVD coatings were deposited on polished (using 1 µm diamond in the last step) ASP 2053 HSS substrates.

Two different metal powders, a water atomized 316L austenitic stainless steel powder and a water atomized Stellite 12 powder, were included in the tests, see Table 1 and Fig. 2. Both powders were tested with and without 1 wt.% internal lubricant in the form of wax. The Stellite powder containing wax also contained 1,4 wt.% of admixed graphite powder for the alloying of the powder.

2.2 Experimental

Before testing the HSS samples (40x20x3 mm) were ground and polished to mirror finish (using 1 μm diamond in the last step). A commercial surface profilometer, Taylor-Hobson Surtronic 4, was used to determine the R_a-values of the samples. The hardness of the HSS samples and the composite hardness, i.e. the as measured hardness, of the PVD-coated HSS were obtained using micro-Vickers hardness indentation and a normal load of 50 g. The thickness of the PVD coatings was obtained from polished cross-sections analyzed in the SEM.

Table I. HSS materials, PVD coatings and metal powders included in the present study.

Material	Chemical composition [weight %]
ASP 2040	1.10 C, 4.2 Cr, 3.0 Mo, 3.3 W, 8.3 V, 1.6 N, bal Fe
ASP 2053	2.48 C, 4.2 Cr, 3.1 Mo, 4.2 W, 8.0 V, bal Fe
PVD CrN	CrN
PVD TiN	TiN
PVD (Ti,Al)N	(Ti,Al)N
PVD WC/C	WC/C multilayer structure
SS 316 L Powder	0.02 C, 17 Cr, 13 Ni, 2.2 Mo, bal Fe
Stellite 12 Powder	1.1 Si, 29.2 Cr, 1.2 Fe, 1.5 Ni, 8.2 W, bal Co, (1.4 C)

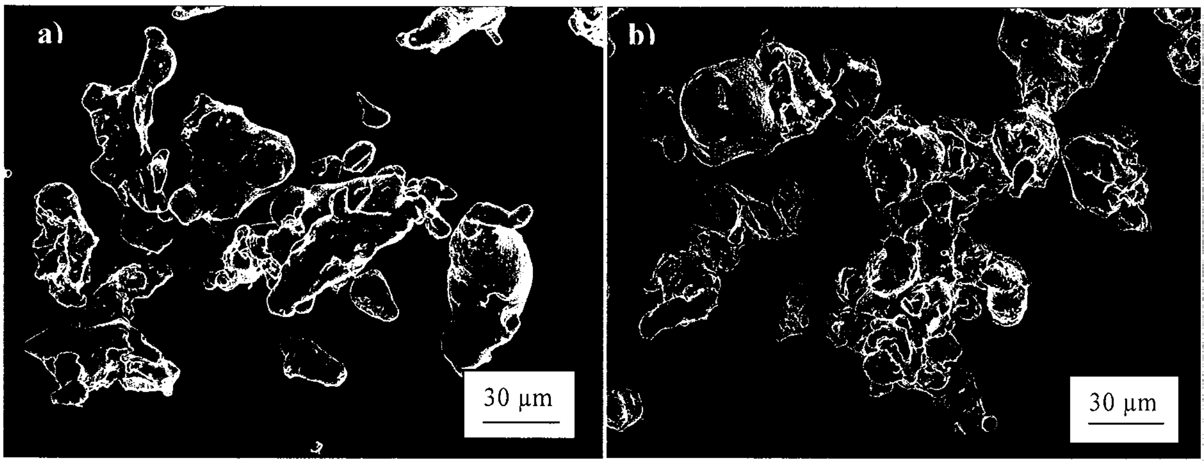

Figure 2. SEM micrographs of the metal powders investigated. Water atomized 316 L stainless steel powder (a) and water atomized Stellite 12 powder (b).

The material transfer tendency between the metal powders and the HSS materials / PVD coatings were evaluated using a CSM Revetest® scratch tester but instead of the Rockwell C diamond stylus commonly used in scratch adhesion experiments a custom manufactured sample holder of aluminum was used, see Figure 3. Prior to the test, the powder to be tested was mounted on the tip of the sample holder of aluminum by simply pressing the tip into a small amount of powder particles poured on the sample surface. In the present study the sample holder had a flat tip with a diameter of 1 mm, see Fig. 3.

Two different test series were performed. In the first test, the possibility to determine a critical normal load for material transfer from the metal powder to the counter surface during a single pass sliding contact was evaluated by gradually increasing the normal load from 1 to 100 N at a loading rate of 50 N/min (sliding distance 15 mm). In the second test, the friction characteristics and material transfer tendencies of the different sliding couples were evaluated during a multi pass linear reciprocating contact using a normal load of 90 N, a sliding velocity of 15 mm/min and a sliding distance of 15 mm. These tests were run until severe material transfer was observed either by a high acoustic emission signal, a high friction coefficient or by observation of the sample surface. Before testing, the uncoated and PVD coated samples were ultrasonically cleaned in acetone, ethanol and methanol in order to ensure that no contaminants on the sample surface would affect the friction. All tests were performed in controlled room temperature (20-22 °C) and relative humidity (35-40 %) and repeated five times. After each test the tip of the sample holder as well as the test sample were studied using SEM/EDS in order to verify that no aluminum from the sample holder had been in contact with the test sample during the test. In the second test, the average friction coefficient, μ_a, during the middle 10 mm distance of each pass was calculated and used as a characteristic friction coefficient for the corresponding pass. It should be noted that no extra lubrication was used in the tests, i.e. the only lubricant active was the internal lubricant of the powder.

3 RESULTS

3.1 Material characterization .

The surface roughness of the uncoated and PVD-coated HSS samples was found to be in the range 0.05 < R_a < 0,1 μm which is typical for the surface roughness of a PM die. This is of importance since it has been shown that in order to be able to distinguish the intrinsic friction characteristics of the different HSS grades and PVD-coatings the surface roughness of the surfaces should be below 0.1 μm [11].

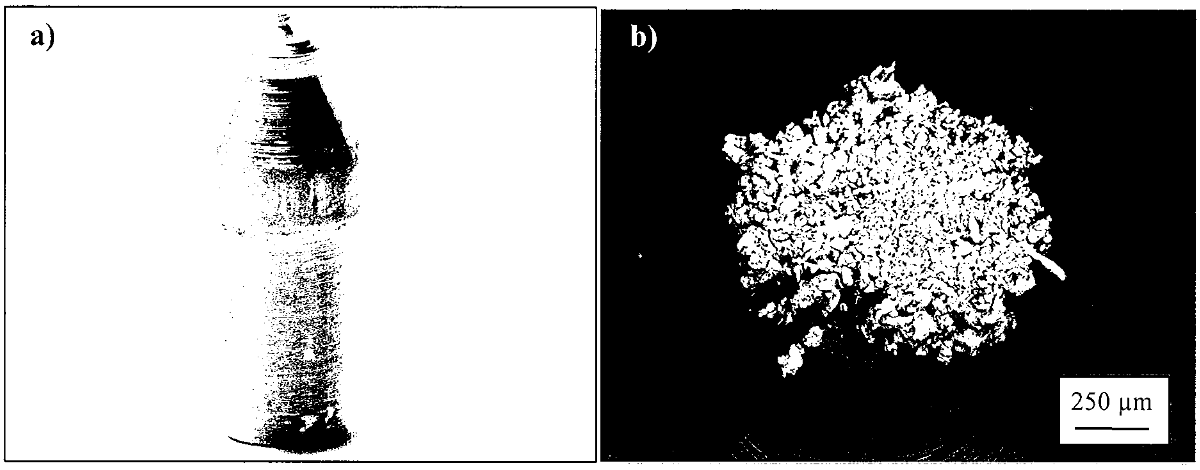

Figure 3. a) Sample holder used in the present study. b) Overview of the flat tip with partly embedded particles.

The hardness values of the uncoated and PVD-coated samples are given in Table II. Each hardness value presented is the mean value of five indentations randomly distributed over the sample surface and σ_{n-1} is used as a measure of the scatter.

Table II. Hardness values of HSS:s and PVD-coatings investigated.

Material	Hardness HV $_{0.05}$ [kg mm^{-2}]	Coating thickness [μm]
ASP 2040	880 ± 10	-
ASP 2053	940 ± 10	-
PVD CrN	2020 ± 180	4.4 ± 0.3
PVD TiN	2210 ± 220	3.8 ± 0.3
PVD (Ti,Al)N	2650 ± 280	4.1 ± 0.5
PVD WC/C	1470 ± 130	2.8 ± 0.3

3.2 Tribological testing

Table III shows the results from the single pass tests using a continuously increasing normal load. The critical normal loads presented in the table correspond to the normal load resulting in a continuous transfer of material from the metal powder particles to the counter material surface as determined by optical microscopy. Although the scatter in the data was relatively high, typically 20% of the given mean values, the results clearly reveal the material transfer characteristics of the combinations investigated. Under unlubricated sliding contact conditions, the only counter material surface not showing material pick-up is the WC/C coating. Furthermore, the tendency to material transfer under these conditions is generally more pronounced and starts at a lower normal load for the austenitic stainless steel powder as compared to the Stellite powder. For both types of metal powder, the nitrogen alloyed ASP 2040 HSS grade display a lower material pick-up tendency as compared with the ASP 2053 HSS grade. The nitride based PVD coatings all show a material pick-up tendency similar, or even worse, to that of the ASP 2053 HSS grade. Under lubricated sliding contact conditions the tendency to material transfer is negligible for all material combinations evaluated in the load range investigated.

Table III. Critical normal loads for the initiation of material transfer from the metal powder to the counter material surface.

Counter material	Critical normal load [N] Stainless steel powder		Critical normal load [N] Stellite powder	
	Unubricated	Lubricared	Unlubricated	Lubricared
ASP 2040	15	>100	30	>100
ASP 2053	<5	>100	15	>100
PVD CrN	<5	>100	<5	>100
PVD TiN	10	>100	10	>100
PVD (Ti,Al)N	<5	>100	10	>100
PVD WC/C	>100	>100	>100	>100

Figure 4 show the friction characteristics of the austenitic stainless steel powder and Stellite powder in contact with the two HSS materials and the PVD WC/C coating, respectively. Except for a lower friction coefficient value for the Stellite powder, the two powders show similar friction characteristics in contact with the two HSS materials, i.e. a rapid increase in friction coefficient with increasing sliding passes for the unlubricated powders and a slower increase in friction coefficient with increasing sliding passes for the lubricated powders. The results indicate that unlubricated powders tend to generate strong adhesive metal-metal contacts during the first sliding pass resulting in material transfer from the metal powder particles to the harder counter surface while this tendency is significantly lower in the case of lubricated metal powders. The same trends were also observed for the three nitride based PVD coatings (not shown in the figures), i.e. these coatings did not show any positive affect in order to reduce the material transfer tendency. For all these combinations the increase in friction coefficient corresponds to an increased tendency to material transfer from the softer metal powder to the harder counter surface. In contrast, the WC/C-coating shows, after an initial peak, a low and stable friction coefficient during the sliding event and long-time testing did not reveal any material transfer / material pick after 300 passes for both unlubricated and lubricated metal powders.

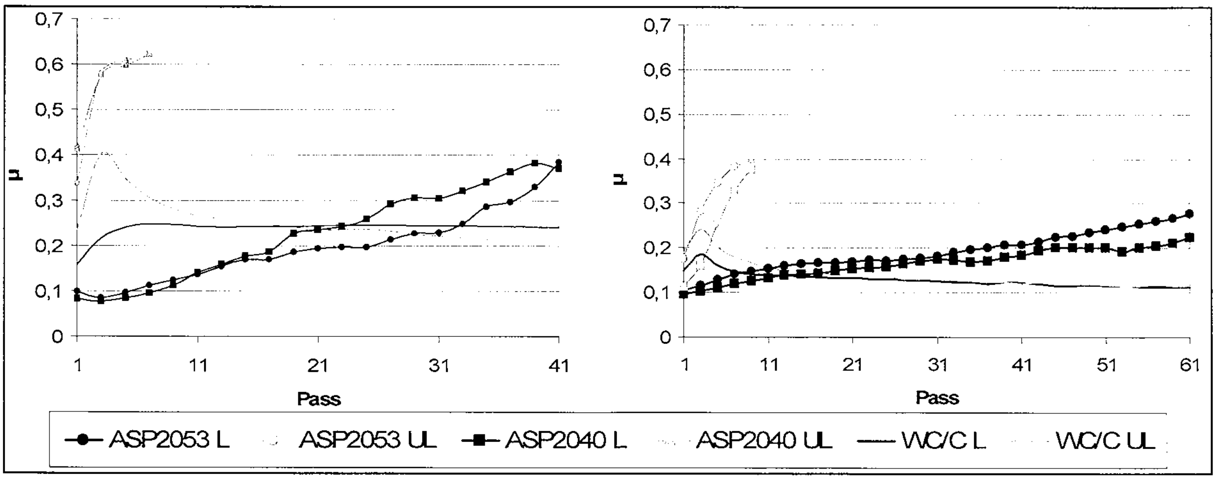

Figure 4. Friction coefficient, μ_a, vs. number of passes for stainless steel (a) and Stellite (b) powder in sliding contact with HSS grades ASP 2040 and ASP 2053 and WC/C PVD coating.

Figure 5 shows the material transfer tendency of unlubricated Stellite powder to the two HSS grades and the TiN and WC/C coatings, respectively, as observed in the SEM. For the two HSS grades and the TiN coating (also true for the CrN and (Ti,Al)N coatings) a significant amount of powder material has adhered to the surface just after a single pass. In contrast, very little powder material has been transferred to the WC/C coating after 300 passes illustrating excellent anti-sticking/low friction properties of this coating. The results from testing against the austenitic stainless steel powder show the same behavior although the amount of transferred powder material is somewhat larger.

4 DISCUSSION

In the present study, the material transfer tendency between two different metal powders, i.e. 316L austenitic stainless steel and Stellite 12, and uncoated and PVD-coated HSS surfaces have been evaluated by a scratch tester using a custom manufactured sample holder for the metal powder particles.

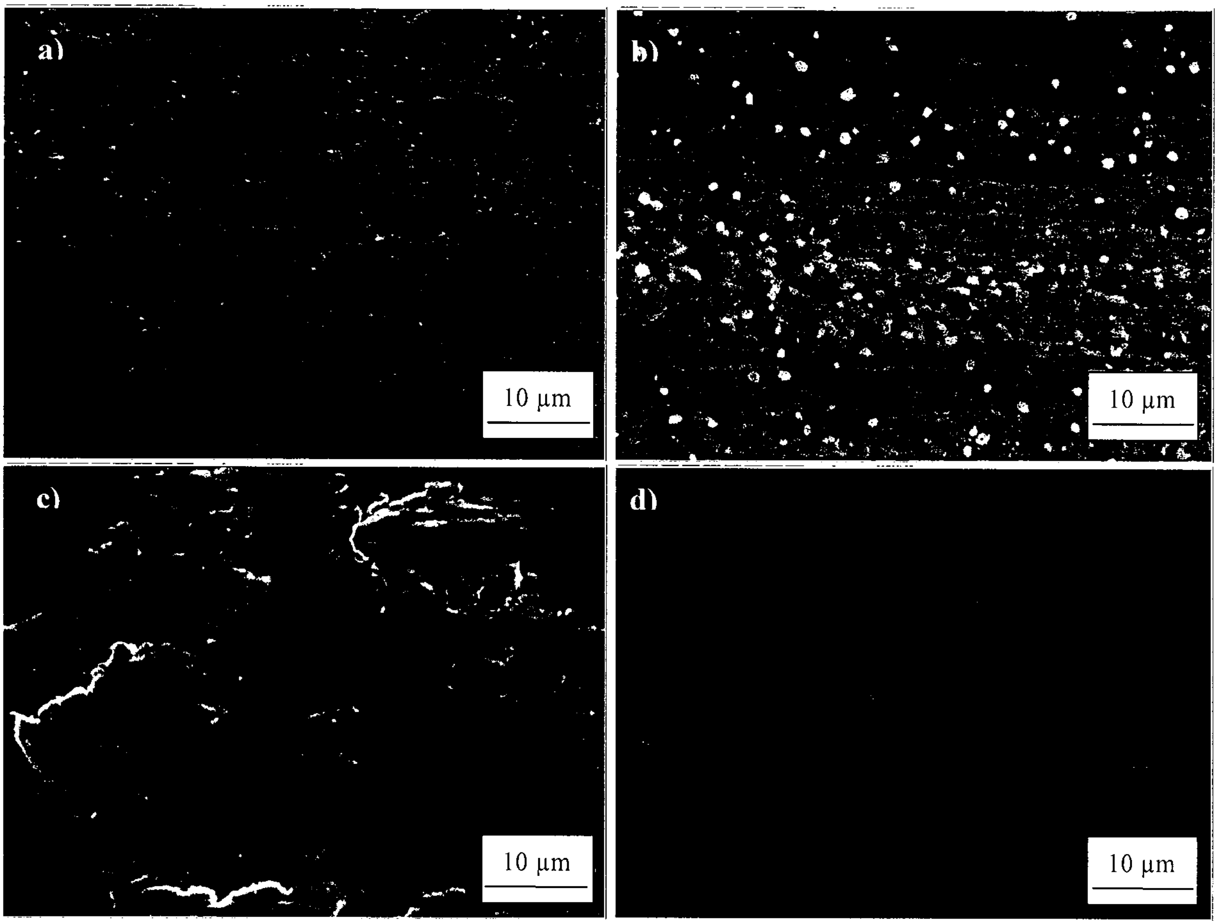

Figure 5. Material transfer tendency from non lubricated Stellite powder to ASP2040 (a), ASP2053 (b), TiN (c) after one single pass and to WC/C (d) after 300 passes as observed in the SEM.

Of the two powders evaluated, the Stellite powder shows a lower friction coefficient and an overall lower material transfer tendency as compared with the austenitic stainless steel powder. This result is in good agreement with previous powder compaction experiments using these two metal powders and is most probably due to the relatively lower shear strength of Stellite as compared to austenitic stainless steel. The results also illuminate the positive effect of internal lubrication in reducing the friction coefficient, i.e. lowering the shear strength at the sliding interface, and thus the material transfer tendency. However, the results show that with increasing sliding passes the internal lubricant is consumed which result in a less effective lubrication and an increased friction coefficient and material transfer tendency. Consequently, although lubricated powders reduce the material transfer tendency and the resulting friction coefficient these can not completely avoid problems caused by material transfer to the tool surfaces during powder compaction.

Although both HSS grades investigated show a significant tendency to material pick-up in sliding contact with the metal powders, the nitrogen alloyed ASP 2040 grade shows a lower material pick-up tendency as compared with the ASP 2053 grade. Consequently, it is believed that nitrogen alloyed ASP 2040 HSS grade shows a potential for minimizing the amount of lubricant in the powder without severe material pick-up problems, especially for less difficult to compact metal powders such as Stellite. Of the PVD coatings evaluated, only the WC/C coating, showing intrinsic low friction and anti-sticking properties, show promising performance also when unlubricated powders are used. In contrast, conventional nitride based PVD coatings such as TiN, (Ti,Al)N and CrN show no positive effect when it comes to reducing the material transfer tendency. This result is in good agreement with earlier results found in the literature [3, 4, 5, 6, 7, 8].

As shown in Figure 4 testing with lubricated powders reduce the material transfer tendency during the early stages of the multi pass testing and material transfer will start to appear first after a critical number of passes depending on the metal powder and the evaluated counter material. Since no additional lubricant has been used in the experiments, the admixed lubricant in the form of wax is gradually broken down during testing and the contact heads towards an unlubricated contact. This differs from the actual powder pressing process where new powder and thus new lubricant is supplied to the contact region for each component that is being pressed. However, the extremely thin lubricant film, typically below 50Å [12], that is produced between the green body and die wall during powder pressing can easily be broken or lost due to a number of reasons. For example, any defects in the die surface can cause the film to be broken and the contact will be unlubricated resulting in material transfer. If this occurs the material transfer process will accelerate at this point eventually causing galling which in turn may result in components of unaccepted surface quality / tolerance or even tool failure. Under such conditions a low friction PVD coating, such as the WC/C coating evaluated in the present study, will certainly increase the reliability of the powder compaction process also when the amount of internal lubricant is low.

5 CONCLUSION

A new approach for evaluating the tribological conditions in powder compaction has been used in order to evaluate the friction characteristics and material transfer tendency between metal powders and potential die materials, including different HSS grades and PVD coatings. The method is based on controlled scratch testing using a commercial scratch tester but instead of the commonly used Rockwell C diamond stylus powder particles are drawn over the surface in a well controlled sliding contact. The main conclusions are:

For all die materials evaluated 316L stainless steel powder shows a higher friction coefficient and a higher material transfer tendency as compared with Stellite 12 powder.

The use of an internal lubricant (wax) significantly reduces the friction coefficient and the material transfer tendency for both types of metal powder. However, an internal lubricant can not completely eliminate the tendency to material transfer from the powder to the counter surface.

Of the HSS grades evaluated, the nitrogen alloyed ASP 2040 shows a lower material pick-up tendency as compared with the ASP 2053 grade.

Of the PVD coatings evaluated, only the WC/C coating, showing intrinsic low friction and anti-sticking properties, shows promising performance also when unlubricated powders are used. In contrast, conventional nitride based PVD coatings such as TiN, (Ti,Al)N and CrN show no positive effect when it comes to reducing the material transfer tendency.

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