



Tribological behaviour of MoS₂-based self-lubricating laser cladding for use in high temperature applications

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ABSTRACT

Many high temperature (HT) forming processes require the use of solid lubricants in order to control friction and reduce wear. In an attempt to eliminate the need for solid lubrication in high temperature sliding applications, nickel-based self-lubricating coatings with the addition of Ag and MoS₂ were prepared by means of laser cladding on stainless steel substrates.

The behaviour of the resulting laser claddings was thoroughly evaluated up to 600 °C, including the oxidation behaviour and reciprocating tribotesting using different counter body geometries (ball and flat pin). The self-lubricating coatings showed lower friction than the unmodified reference alloy at all tested temperatures, in addition to a significant microstructural stability after prolonged exposure at high temperatures. The addition of solid lubricants to the claddings was also found to be beneficial in terms of the counter body wear at HT, as no material loss could be measured for the bearing balls after testing at 600 °C against the self-lubricating claddings, despite the significant softening experienced by AISI 52100 bearing steel at HT.

1. Introduction

The use of lubricants during metal forming processes is a common procedure, as it helps not only to reduce tool wear but also to control friction to low and stable values, as a small amount of plastic deformation preventing surface wrinkling of the blank piece is necessary to ensure the quality of the finished product [1]. However, the use of lubricants in such industrial processes involves significant environmental and health hazards, not to mention the related cleaning steps [2,3] and increased disposal costs [4] as in recent years more strict regulations are being implemented. Oils and greases have other significant disadvantages as they thermally degrade if used in forming processes involving temperatures above 300 °C, like for instance hot stamping. For these reasons, the development of new lubrication methods is considered to be beneficial for HT applications and has the potential to mitigate costs and decrease environmental hazards while at the same time ensuring the quality of the produced parts. However, such an approach is not devoid of problems. In recent years, there has been an increased interest in vanadium-based self-lubricating materials for use in demanding applications such as metal cutting or aerospace [5,6]. However, vanadium-based compounds also pose health risks due to their toxicity. A reported solid lubricant like V₂O₅ can detrimentally affect the respiratory function [7], while other promising compounds

like vanadates have been reported to interfere with key cellular processes [8], so their implementation in industrial applications should be carefully considered.

In this context, low-toxicity solid lubricants like silver and MoS₂ have been regarded as a better choice for hot forming processes. In fact, they have been the subject of many studies in recent years due to their self-lubricating behaviour at high temperature (HT) [9]. Silver is an effective lubricant at HT whose main mechanisms is based on its low shearing strength and its diffusion to the sliding surface at HT [10,11]. The lubricous role of transition metal dichalcogenides (TMDs) like MoS₂, on the other hand, is based on their layered microstructure. Such layers can align in the direction of motion and slide easily over each other, decreasing friction and wear up to temperatures of 400 °C [12]. The combined use of silver and MoS₂ in composite materials is also of interest as it can lead to the in-situ formation of lubricous silver molybdates at temperatures between 300 and 600 °C. The crystal structure of molybdates such as Ag₂MoO₄ and Ag₂Mo₂O₇ has been described to be layered as well [9,13], with the Ag-O and O-Ag-O interlayer bonds being particularly weak and thus shearing easily and providing effective lubrication at HT.

New lubrication strategies as the ones previously described are of significant interest for industrial applications like hot metal forming, as severe contact conditions are prevalent during such processes. In this

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context, decreased tool wear in addition to low and stable friction are beneficial in such applications to ensure the surface quality of the resulting product. Thick layers with increased wear resistance can be deposited by means of laser cladding, a manufacturing process which also allows for the reworking and repairing of high-value industrial components. This technique involves the melting of a precursor in powder form by means of a laser beam to produce coatings with low defect density [14], reduced dilution [15,16] and at the same time excellent metallurgical bonding with the substrate [17]. In an effort to control the friction of the resulting claddings, the addition of different classes of solid lubricants to the base powder has been studied in recent years [18–20], showing promising results for HT applications. However, the high temperatures reached during laser cladding can lead to the thermal degradation of TMDs like MoS₂ and WS₂ solid lubricants [21–23]. In addition to the chemical composition and the preparation technique, the chosen testing configuration can also influence the tribological behaviour of self-lubricating materials. The lab-scale simulation of tribological systems of industrial interest is done in many cases using Hertzian point contacts like the well-known ball-on-disc configuration. Such geometries have significant advantages as non-conformal contacts allow for an easy alignment of both samples, in addition to the generation of measurable wear scars in a comparatively short testing time due to the severe contact conditions. However, despite its popularity for tribotesting, point contacts involve unrealistic assumptions and can lead to wear mechanisms different from those observed in actual applications. Interestingly, it has even been reported that there are no industrial applications featuring the pure sliding of point contacts [24]. Additionally, the initial contact pressures for non-conformal geometries like ball-on-disc can be very high and are usually linked to severe running-in wear during the initial stages of testing. As the contact area increases due to wear, contact pressure quickly decreases leading to changes in wear mechanisms. However, such a behaviour is unrealistic as industrial applications are usually designed to operate under constant pressures, and thus line and area contact geometries should be considered instead for more realistic tribotesting. Point contacts can be used for screening tests due to accelerated wear and high repeatability, but closer simulation of real applications must be performed under testing configurations with lower contact pressures.

In the present study, laser claddings with the addition of silver and MoS₂ have been prepared and tribologically characterised up to 600 °C, aiming at their implementation in metal forming applications like hot stamping. To this end, different testing configurations have been chosen: Hertzian point contacts in addition to lower pressure flat contacts expected to be closer to metal forming applications. The oxidation resistance and microstructural stability of the claddings have also been studied after prolonged exposure to HT, as it is an important property for their prospective use in metal forming.

2. Experimental

2.1. Materials preparation

For the present study, the deposition of the claddings was performed by means of a direct diode laser. This technique offers significant advantages like the single-pass melting of the precursor material, an excellent metallurgical bonding with the substrate [17] and the low defect density of the resulting claddings [14].

A NiCrSiB commercial powder supplied by Castolin Eutectic was chosen as the base material of the cladding alloy, with a chemical composition of 0.2 C, 4 Cr, 1 B, 2.5 Si, < 2 Fe and 1 Al (in wt. %) with a grain size between 50 and 150 µm. The addition of boron and silicon lowered the melting point of the mixed powder, improving the deposition of this nickel-based alloy [25]. Ag powders to be added as solid lubricants were supplied by Goodfellow, with a maximum particle size of 45 µm MoS₂ powder was provided by Tribotec GmbH with a particle size between 5 and 75 µm. Table 1 lists the chemical composition of the

Table 1
Solid lubricant content for the deposited coatings.

Claddings	Solid lubricant content (wt. %)		RT hardness [HV1]	Ra (µm)
	Ag	MoS ₂		
Reference	0	0	393 ± 13	0.09 ± 0.03
10 MoS ₂	0	10	392 ± 15	0.10 ± 0.02
5 Ag – 10 MoS ₂	5	10	398 ± 5	0.10 ± 0.03

claddings deposited during this study, including the reference unmodified NiCrSiB cladding in addition to several self-lubricating hard-facings with silver and MoS₂. This was aimed at investigating the role of both solid lubricants on the microstructure, hardness and HT tribological behaviour of the resulting coatings.

The coatings were deposited on grade 1.4301 stainless steel plates to prevent the oxidation of the substrate during laser cladding and HT tribotesting. Prior to laser cladding, the plates were rinsed with ethanol and sandblasted with silica sand in order to improve the adhesion of the resulting coatings. The Ni-based powder and the solid lubricants were mechanically mixed using ethanol as the binder, and spread over the stainless steel substrate. This mixture was heated in an oven at 100 °C during 1 h in order to ensure the evaporation of ethanol before cladding, which was performed under a protective argon atmosphere to prevent the oxidation of the resulting claddings. The choice of this procedure over other similar techniques such as powder flow or wire feeding led to a simpler cladding deposition process.

A thorough description of the laser cladding parameters used during the deposition process such as beam speed and input power together with a detailed description on the optimum selection of solid lubricant content is given in a previous publication by the authors [26].

Sample preparation prior to testing included the machining to a size of 12.6 × 12.6 × 4.7 mm³, subsequent manual grinding using #360 and #600 grit sizes SiC- abrasive papers, and rotating the samples during the last step to remove any directionality in the observed surface topography. Roughness R_a prior to testing was measured using a Zygo New View 7300 3D optical profiler and was found to be ~0.1 µm for all of the chosen claddings. All specimens were ultrasonically cleaned in petroleum ether and rinsed with acetone before tribological tests.

2.2. High temperature oxidation tests

HT oxidation tests have been described for self-lubricating materials in the available literature [27]. In the present study, such tests were chosen to obtain further information on the behaviour of the deposited laser claddings at high temperatures, especially their thermal stability and the diffusion of silver to the sample surface, a process which may lead to lubricant depletion [28] and in some cases to coating collapse due to increased porosity [29]. To this end, samples of both the unmodified nickel-based alloy and 5 Ag – 10 MoS₂ were chosen, and heated up to 600 °C in an induction oven in air. For the reference material and 5 Ag – 10 MoS₂ two samples were used: the first one was tested for 48 h and the second one for 100 h. Further characterisation of the tested samples was done by means of optical microscopy with an emphasis on the resulting microstructure of the claddings and SEM/EDS to characterise the resulting oxide layers both in terms of morphology and chemical composition.

2.3. High temperature reciprocating tests

The tribological behaviour at HT of the laser claddings was studied using an Optimol SRV friction and wear tester. An upper counter body sample was loaded against a stationary cladding sample by means of a spring deflection mechanism during the test. The upper sample oscillated by means of an electromagnetic drive while the lower flat sample

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