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Obdelovalnost vroče stisnjenih samomazalnih kompozitov Fe-MnS in Fe-MoS_2

Machinability of Hot-Pressed Self-Lubricating Fe-MnS and Fe-MoS_2 Composites

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Poleg standardnih triboloških preskusov, s katerimi merimo koeficient trenja in obrabo drsnih stikov pod natančno določenimi pogoji preskušanja, lahko kot merilo za samomazalnost izdelanih kompozitov uporabimo tudi izmerjene velikosti rezalnih sil. Te podatke se običajno uporablja kot merilo za obdelovalne karakteristike določenega materiala. V prispevku so predstavljeni rezultati sinteze Fe-MnS in Fe-MoS_2 kompozitov, izdelanih s postopkom vročega stiskanja. Njihove mikrostrukturne lastnosti so povezane z vrednostmi rezalnih sil, izmerjenih pri postopku vzdolžnega struženja.

(Ključne besede: kompoziti samomazalni, kompoziti kovinski (Fe), obdelovalnost, sile rezanja)

In addition to the standardised tribological tests for determination of the coefficient of friction and wear at the sliding contacts under exactly defined testing conditions, the measured magnitudes of cutting forces can also be used as a measure for the self-lubrication of the composites produced. Such data are most commonly used as a measure for the machinability characteristics of a particular material. The paper presents the results of the synthesis of Fe-MnS and Fe-MoS_2 composites produced with the process of hot-pressing. Their microstructural properties are related with the values of the cutting forces which were measured during the longitudinal turning process.

(Keywords: self lubricating composites, Fe-based composites, machinability, cutting forces)

0 UVOD

Pod pojmom obdelovalnost razumemo lastnost materiala, da ga je mogoče obdelovati oziroma predelovati. Definicija je zelo široka in ne pomeni le obdelovalnost materiala pri obdelavi z odrezovanjem, ampak tudi pri drugih načinih mehanske obdelave. Včasih obdelovalnost glede na odrezovanje imenujemo kar odrezovalnost. Širše lahko ta pojem opredelimo kot sposobnost materiala, da ga je sploh mogoče odrezovati. Taka definicija je sicer na videz jasna, vendar jo je težko podrobneje določiti, zlasti pa meriti [1]. Odrezovalnost je kljub temu zelo pomembna lastnost materialov. Upoštevati bi jo morali že konstrukterji, saj bo le tako mogoče izdelati izdelke z zahtevano kakovostjo in čim bolj poceni. Po ožji definiciji je bolje obdelovalen tisti material:

- ki ga lahko obdelujemo,
- pri katerem je obstojni čas orodja daljši,
- pri katerem so manjše odrezovalne sile,
- pri katerem dobimo boljšo kakovost obdelane

0 INTRODUCTION

By the concept the machinability we understand the material property for machining or forming. This definition, rather a short one, means not only the machining characteristics of the materials obtained with the cutting process but also by other methods of mechanical material processing. Sometimes the machinability with regard to the cutting process is simply denoted as cutting-ability. In the broad sense this notion can be defined as the ability of the material to be cut at all. Such a definition is apparently clear but is difficult to determine in greater detail, and especially difficult to measure [1]. Cutting-ability is, however, one of the most important characteristics of the material. It must be considered already by the designers because only in such a way can products with the prescribed quality and prices as lower as possible be produced. According the close definition, better machinability characteristics are demonstrated by those materials:

- which can be more easily machined,
- where the tool-life is longer,
- where the cutting forces are lowest,
- where a better machined surface is obtained,

površine,

- pri katerem dobimo ugodnejšo obliko odrezkov,
- pri katerem dosežemo večjo natančnost obdelave.

Vsako od naštetih stališč pomeni pomembno okoliščino pri določanju obdelovalnosti za določen material. S številnimi preskusi so skušali dobiti stalne vrednosti ali določiti parametre, s katerimi bi lahko ugotavljali obdelovalnost posameznih materialov [2]. V tovrstno eksperimentalno delo je bilo v preteklosti vloženih mnogo naporov širom po svetu. Postopke določanja obdelovalnosti so poskušali poenotiti do te mere, da bi bilo mogoče oblikovati skupne baze podatkov obdelovalnosti za različne kombinacije obdelovanec – orodje ([3] in [4]). Poleg tega, da je obdelovalnost v največji meri podatek, potreben tehnologu v proizvodnji, pa so ti podatki lahko v pomoč tudi razvijalcem novih materialov, saj lahko iz obdelovalnih karakteristik sklepajo o različnih lastnostih materialov. Takšen specifičen primer so tudi samomazalni materiali, kjer določeni dodatki vplivajo na obdelovalnost, podobno kakor zunanja hladilno-mazalna sredstva.

- where a favourable chip shape is obtained,
- where better accuracy of the machining is obtained.

Each of the listed standpoints means an important circumstance in determining the machinability of the prescribed material. Through numerous experiments, researchers have tried to obtain some constant values or have determined parameters by which the machinability of particular materials can be established [2]. In the past, great efforts have been invested all over the world in such experimental work. Researchers have tried to determine the machinability procedures to such an extent that the common data base on the machinability parameters for different combinations of workpiece material-tools can be formed ([3] and [4]). Machinability provides mainly the information which is needed the technologist in production, in addition these data can also be help for the developers of new materials; from various machinability characteristics they can establish different characteristics of the materials. Such a specific case is also demonstrated in the self-lubricating materials where particular ingredients influence into the machinability characteristics in a similar way as external cooling-lubricated liquids.

1 OSNOVNE ZNAČILNOSTI TRDNIH MAZIV IN SAMOMAZALNIH KOMPOZITOV

V nekaterih primerih kot maziva uporabljamo trdne materiale z majhnim koeficientom trenja. Trdno mazivo je lahko na površini, bodisi v obliki tanke plasti ali kot sestavina kompozitnega (samomazalnega) materiala. Takšna izvedba mazanja je cenejša in manj zahtevna za vzdrževanje kot mazanje s tekočimi mazivi (olji ali mastmi). V določenih primerih je mazanje s trdnim mazivom edina mogoča, oziroma še sprejemljiva izvedba (strojni deli v proizvodnji hrane, vesoljska plovila, sateliti, vakuumske naprave, deli jedrskih reaktorjev itn.), zaradi higienskih razlogov, visokih delovnih temperatur, nevarnosti odparevanja ali težavah pri stalnem dovajanju tekočega maziva na površine, ki so v tribološkem stiku.

V določenih razmerah imajo nekatere trdne snovi z lamelarno strukturo majhen koeficient trenja. Zato so zanimive za uporabo kot trdna maziva. Med materiali z lamelarno strukturo sta najbolj poznana grafit in molibdenov disulfid (MoS_2). Pri MoS_2 so plasti atomov žvepla medsebojno vezane s šibkimi, Van-der-Waalsovimi vezmi, medtem ko so vezi med atomi žvepla in Mo kovalentne in zato močne. Rezultat je plastna struktura, katere posledica je značilno obnašanje MoS_2 v tribološkem stiku z drugimi materiali. Majhen koeficient trenja obeh materialov pripisujemo njuni lamelarni strukturi in šibki vezi med bazalnimi ravninami, čeprav to ne pomeni, da bodo vsi materiali s podobno strukturo

1 BASIC CHARACTERISTICS OF SOLID LUBRICANTS AND SELFLUBRICATING COMPOSITES

For some special applications, solid materials with a low friction coefficient are used. In this case a solid material (lubricant), either in the form of a thin layer, or as a component of composite self-lubricating material on the sliding surface is present. This type of lubrication is cheaper and less complex for maintenance in comparison with oil or grease lubrication. In some cases (e.g.: machine and/or structural parts in food production, for space shuttles, satellites, vacuum equipment, in nuclear power plants etc.), the solid lubrication is the only possible and acceptable solution because of either hygienic reasons, high working temperatures, excessive evaporation of liquid lubricant or difficulties in assuring a continuous supply of liquid lubricant on the surfaces which are in tribological contact.

Under certain conditions of application, some solid substances with lamellar structure have a low coefficient of friction. Among these materials, graphite and molybdenum disulphide (MoS_2) are well known and widely used. In the molecular structure of MoS_2, the layers of sulphur atoms are bound together by weak Van-der-Walls forces, while the atomic bonds between sulphur and Mo atomic layers are covalent and therefore strong. The resulting sandwich structure produces the characteristic behaviour of MoS_2 when it is in tribological contact with other materials. The low friction coefficient of both materials is attributed to their lamellar structures and to weak inter-planar bonding. However, this does not imeli nizek koeficient trenja. Prehodni kovinski dihalogenidi imajo podobno strukturo kakor MoS_2. Tako disulfidi (NbS_2, TaS_2, WS_2), diteluridi in diselenidi teh kovin (na primer $MoTe_2$ ali $MoSe_2$) delujejo kot trdna maziva. Vendar se nobeden od teh materialov ne uporablja tako pogosto kakor MoS_2. Nekatere anorganske spojine so kljub temu, da nimajo lamelarne strukture, uporabne kot trdna maziva, še posebej pri visokih temperaturah, ker imajo majhno strižno trdnost. Tako so CaF_2 in BaF_2, PbO in PbS ter B_2O_3 materiali, ki jih lahko uporabimo kot trdna visoko temperaturna maziva bodisi v obliki tanke plasti ali kompozita.

Koeficient trenja MoS_2 je močno odvisen od atmosfere, v kateri se nahaja. MoS_2 ima majhen koeficient notranjega trenja in zato najnižji koeficient trenja v vakuumu. S povečano vsebnostjo kondenziranih par se koeficient trenja pri MoS_2 v nasprotju z grafitom zvečuje. Bolj, ko je čist, manjši ima koeficient trenja ([5] in [6]), zato je primeren kot trdno mazivo, predvsem v vakuumu in nevtralni atmosferi. Poleg tega je njegova termodinamična stabilnost v zraku in drugih sredstvih, ki vsebujejo vlago, relativno slaba in nastanek abrazivnih oksidov (MoO_2) povečuje koeficient trenja. Poleg nosilnega učinkovanja je MoS_2 znan tudi po tem, da učinkuje tribokemično. Na stiku z drsečo se jekleno površino nastaja deloma zelo tanka ločilna plast FeS ali bolje rečeno zapletenih sulfidov, katerih dobre drsne lastnosti so znane.

Za samomazalne kompozitne materiale je značilno, da v kovinski (sl. 1) ali polimerni (redkeje keramični) matrici oziroma njegovi tanki površinski plasti vsebujejo enakomerno razporejene drobne delce trdnega maziva. Najbolj znani samomazalni kompozitni materiali, ki vsebujejo 5 do 15 masnih odstotkov MoS_2, so polimerni materiali na osnovi teflona ali poliamida ([7] in [8]). Samomazalni kompoziti s kovinsko matrico, ki vsebujejo MoS_2, imajo matrico, sestavljeno predvsem iz neželeznih kovin (Cu, Al, Ni) [5], ki ne reagirajo z MoS_2. Nekateri ([6] in [8]) omenjajo tudi kompozite z matrico na osnovi žlahtnih kovin (Ag) in kovin z visokim tališčem (Mo, W, Ta ali Nb) predvsem za strojne dele vesoljskih naprav (Mariner, Apollo, Pegasus itn.). Vsi ti materiali so večinoma izdelani s postopki metalurgije prahov (PM) ([10] in [11]).

V navzočnosti nekaterih standardnih zlitinskih elementov jekla (Cr, Mn), pride lahko med sintezo kompozita na osnovi Fe do razpada MoS_2 in nastanka termodinamično stabilnejših sulfidov. Zato je s standardnim postopkom PM (sintranjem) težko, oziroma praktično nemogoče izdelati kompozit na osnovi Fe, ki ima enakomerno razporejene delce MoS_2. Ena od možnosti je hladno dinamično zgoščevanje (eksplozijsko zgoščanje) ([10] do [12]) prašne mešanice jeklenih in delcev MoS_2. Vendar so jekleni delci, ki so izdelani s postopkom atomizacije

mean that all materials with similar atomic structure exhibit solid lubrication properties. Transition metal dichalcogenides have analogous structure to MoS_2. Disulphides (NbS_2, TaS_2, WS_2), ditellurides and diselenides of these metals (for example: $MoTe_2$ and $MoSe_2$) act as solid lubricants. However, none of these materials is used as widely as MoS_2. Certain inorganic compounds have lubrication properties at high temperatures although they do not have lamellar structure because of their low shear strength. Thus, CaF_2 and BaF_2, PbO and PbS, as well as B_2O_3 are materials which can be applied as high temperature solid lubricants in the form of a thin layer or metal/ceramic based composite.

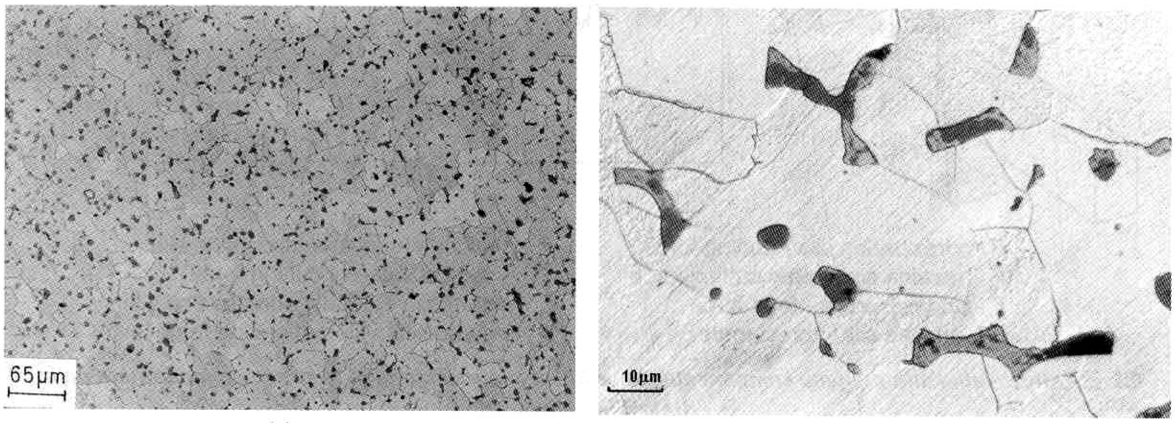
The friction coefficient of MoS_2 strongly depends on the atmosphere. MoS_2 has very low internal friction and therefore the lowest friction coefficient in vacuum ([5] and [6]). The friction coefficient of MoS_2 increases with increased content of condensed vapours. Therefore, it is a very appropriate solid lubricant for application in vacuum or inert gas atmosphere. The thermodynamic stability of MoS_2 in air and humidity is low and the formation of abrasive oxides (MoO_2) increases the friction coefficient. It is well known that MoS_2 besides having a structural effect, acts also tribochemically. In contact with sliding steel surfaces it forms a very thin separating layer of FeS or complex sulphides with excellent sliding characteristics.

The characteristic of self-lubricating composites is fine and uniform dispersion of solid lubricant particles in metal (Fig. 1), ceramic or polymer matrix. Gradient material with such a thin composite surface layer can also be synthesised. The most popular self-lubricating MoS_2 based composite materials are polymer composites with 5-15 mass % of MoS_2 in PTFE (polytetrafluorethylene) or Polyamide matrix ([7] and [8]). Metal based self-lubricating MoS_2 composites usually have a metal matrix based on light (Cu, Al, Ag-alloys) metals [5], which do not react with MoS_2 during high-temperature synthesis. For the machine or structural parts of spacecraft applications (Mariner, Apollo, Pegasus etc.), the composites with noble metals (Ag) or refractory (Mo, W, Ta or Nb) metals based matrix are also mentioned elsewhere ([6] and [8]). All these materials are mostly produced by powder metallurgy routes (PM) ([10] and [11]).

In the presence of some standard alloying elements (Cr, Mn) of steels, the decomposition of MoS_2 and the formation of thermodynamically more stable sulphides can occur during the high temperature synthesis of a Fe-MoS_2 composite. Therefore, it is very difficult or practically impossible to prepare a Fe based MoS_2 self-lubricating composite by the conventional PM practice (sintering). One possibility is dynamic (explosive) compaction ([10] to [12]) of steel and MoS_2 particles powder mixture. However, steel particles prepared by water or gas at-

[13] zelo trdi. Zato je prašno mešanico na ta način težko popolnoma zgostiti. Druga možnost so postopki zgoščevanja pri povišanih temperaturah, vendar mora potekati zgoščevanje dovolj hitro, da ne prihaja do nezaželenih reakcij. Enoosno vroče stiskanje je postopek, ki bi morda lahko omogočal sintezo kompozitov Fe-MoS_2, saj poteka relativno hitro, preprosto in zaradi pomoči tlaka pri nižjih temperaturah kot sintranje. Zato smo s tem postopkom poskušali izdelati kompozit Fe-MoS_2. Za primerjavo smo s postopkom vročega stiskanja izdelali tudi kompozit Fe-MnS, saj se manganov sulfid (MnS) najpogosteje uporablja kot dodatek materialom, katerih obdelovalnost je problematična [14]. Hkrati pa se v nekaterih materialih pojavlja kot mazivo [15] za zmanjšanje trenja med površinami, ki so v tribološkem stiku. Zaradi poroznosti in trdih oksidnih filmov [16] ter s tem povezane neenakomerne (izmenične) obremenitve rezalnih orodij so še posebej težko obdelovalni materiali PM.

omisation are very hard [13] and in this way the powder mixture cannot be compacted to full density. Anther possibility is densification of powder mixture at elevated temperatures, rapid enough to avoid undesired reactions. Uniaxial hot compaction is a fast, simple and pressure-assisted powder consolidation process. Therefore, it might make it possible to synthesize the Fe-MoS_2 composite. In the present paper the synthesis of Fe-MoS_2 by hot pressing is presented. For comparison, a Fe-MnS composite is also prepared. The manganese sulphide (MnS) is the most frequently used addition to the materials with poor machinability[14]. Additionally, it is used as a solid lubricant in some special materials [15] for the reduction of friction between sliding surfaces. In addition, the PM materials are also materials with poor machinability because of their porosity and present oxide films [16] which produces intermittent non-uniform loading of cutting tools and their excessive wear.



(a) (b)

Sl. 1. *Metalografski posnetek vroče stisnjenega kompozita Fe-MnS oziroma Fe-MoS_2 z dobro vidno enakomerno porazdelitvijo oksisulfidnih delcev v feritni osnovi – a) 1,5 masnih % MnS in b) 1,5 masnih % MoS_2*

Fig. 1. *Micro-graph of a hot pressed Fe-MnS and Fe-MoS_2 composite with well visible uniform distribution of fine oxysulphide particles in ferrite matrix – a) 1.5 mass % of MnS and b) 1.5 mass % of MoS_2*

2 REZALNE SILE KOT MERILO ZA OBDELOVALNOST

Za zagotavljanje zadostnih napetosti in deformacij v rezalni coni za tvorbo odrezka mora orodje pritiskati na material z zadosti veliko silo. V strižni coni deluje na orodje enako velika reakcijska sila, ki na površini stika odrezka in obdelovanca obremenjuje rezalni rob orodja. Poleg materiala obdelovanca na velikost in smer odrezovalne sile vpliva še vrsta drugih dejavnikov (rezalni parametri, geometrična oblika orodja, obraba orodja, obdelovalni stroj itn.). Takrat, ko njihov vpliv poznamo ali ga poskušamo obdržati v določenih mejah, so lahko izmerjeni podatki o velikosti rezalnih sil dobro merilo za oceno lastnosti določenih obdelovanih materialov in zato tudi eden od največ

2 CUTTING FORCES AS A CRITERION FOR THE MACHINABILITY

To assure adequate stresses and deformations at the cutting zone for the chip formation the tool needs to press the material with sufficient cutting force. At the cutting zone the same magnitude forces react to the tool loading the cutting edge of the tool at the chip - workpiece interface. Beside the workpiece material, numerous different factors (cutting parameters, geometrical shape of the tool, tool wear, machine tool, etc.) also influence the magnitude and direction of the cutting force. When their influence is known, or retained within the prescribed limits, the measured data on the cutting force magnitudes can be a good measure for the assessment of the characteristics of particular materials and uporabljanih meril za ocenjevanje obdelovalnosti materialov.

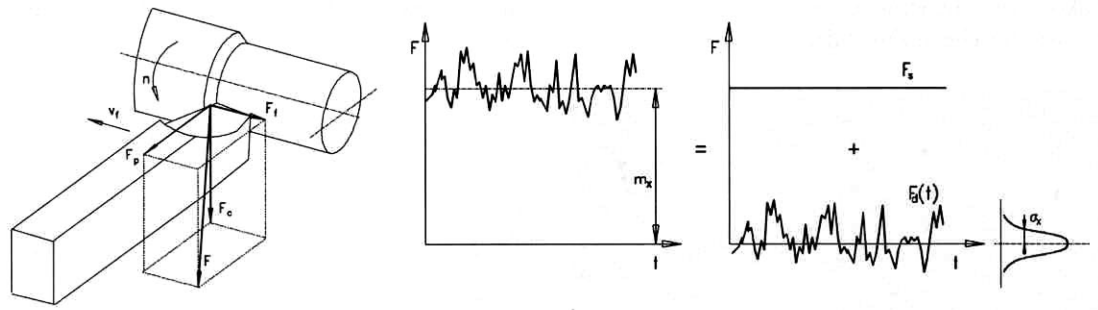
Skupno odrezovalno silo običajno razstavimo na komponente, ki jih lahko merimo: rezalno, podajalno in odrivno silo (sl. 2). Če zapisujemo časovno odvisnost odrezovalne sile ali njene posamezne komponente pri nespremenjenih obdelovalnih razmerah, ugotovimo, da se njihove velikosti s časom spreminjajo. V primeru, ko ta odvisnost poteka kot ustaljen naključni proces, lahko izračunamo njene statistične parametre - povprečno vrednost m_x (statična vrednost sil F_s) in standarno deviacijo σ_x (dinamična vrednost sil F_d) – sl. 2.

Meritve sil pri odrezovanju so posledica problemov, ki se pojavljajo pri analitičnem določanju komponent odrezovalne sile. Ker je nemogoče meriti komponente odrezovalnih sil v njihovih izhodiščih,

therefore also one of the most used criteria for the assessment of the machinability of the materials.

The whole cutting force is usually exhibited by the three components which can be more easily measured: main cutting, feed and thrust force (Fig. 2.). When the dependence of the cutting force or its particular component under unchangeable cutting conditions is registered as a time series we can establish that the values change during time. In cases when the record is in the form of a steady random process we can calculate its statistical parameters - mean value m_x (statical value of the forces F_s) and standard deviation σ_x (dynamical value of the forces F_d) - Fig. 2.

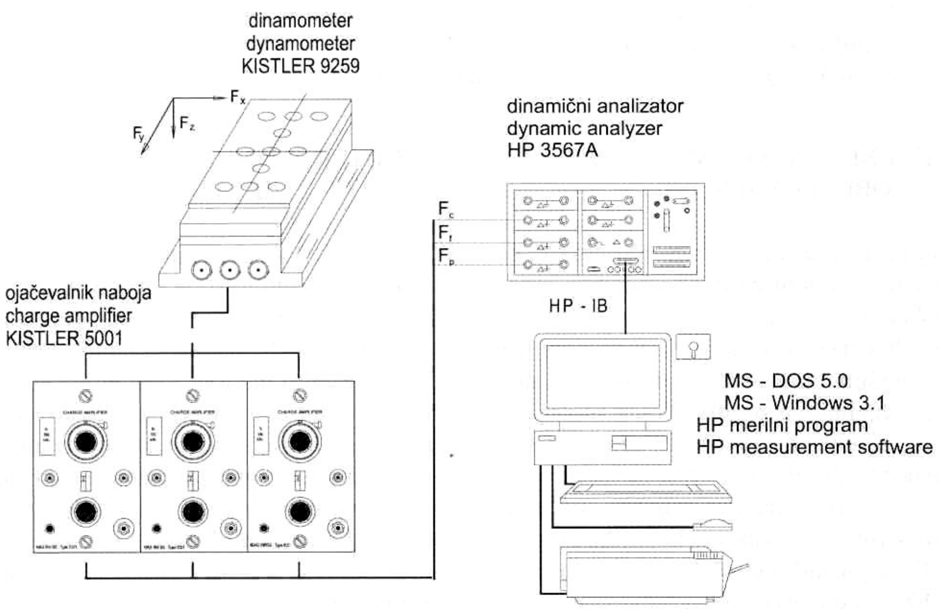
Measurements of the forces are the consequence of the problems which occur in analytical determination of the cutting force components. As it is



F - odrezovalna sila - cutting force
F_c - rezalna sila - main cutting force
F_f - podajalna sila - feed force
F_p - odrivna sila - thrust force
F_s - statična vrednost odrezovalne sile - statical value of cutting force
F_d - dinamična vrednost odrezovalne sile - dynamical value of cutting force

Sl. 2. *Odrezovalna sila in njene komponente pri vzdolžnem struženju in primer zapisa njene statične in dinamične vrednosti*

Fig. 2. *Cutting force and its components at longitudinal turning and the example of the time series record of its statical and dynamical value*



Sl. 3. *Merilni sistem za merjenje komponent odrezovalne sile pri struženju*

Fig. 3. *Measurement system for measurement the cutting force components for the turning processes*

merimo njihove odzive v določeni oddaljenosti od rezalnega robu. Zato potrebujemo merilni sistem, s katerim lahko opravljamo pravilne meritve neodvisno od položaja prijemanja sile ali momenta. Namen razvoja merilnega sistema je industriji zagotoviti uporabne raziskave s preprostimi in učinkovitimi merilnimi sistemi. Na sliki 3 je prikazan merilni sistem za merjenje komponent odrezovalne sile za postopke struženja. Piezoelektrični dinamometer spremeni mehanski signal sile v električni signal, tega okrepimo z ojačevalnikom naboja. Frekvenčni analizator je potreben zapisni del verige zaradi dinamične oblike poteka komponent odrezovalne sile in kratkih rezalnih časov rezalnega robu. Digitalizirani časovni zapis prenesemo in ovrednotimo na osebnem računalniku, na katerem z uporabo ustrezne programske opreme tudi izračunamo statične in dinamične vrednosti komponent odrezovalnih sil.

3 PRIPRAVA VZORCEV IN TESTIRANJE OBDELOVALNOSTI

Izhodni kovinski železov prah smo pripravili s postopkom vodne atomizacije[13]. Po atomizaciji smo izdelani železov prah 3 ure mehko žarili v suhem vodiku pri 850 °C v laboratorijski cevni peči in s tem znižali trdoto prašnih delcev na vrednosti sprejemljive za hladno zgoščevanje. Po taljenju vložka in izdelavi prahu smo izvedli nadzor kemijske sestave z optičnim emisijskim spektrometrom ARL, tip 3460 Metals Analyzer, ZDA. Izdelanemu Fe prahu smo določili osnovne fizikalno-kemijske in morfološke lastnosti (oblika in velikostna porazdelitev delcev, tekočnost, stisljivost itn.) [18]. Za pripravo mešanic z različnim deležem MoS_2 in MnS smo uporabili komercialno dosegljive laboratorijsko čiste surovine (MoS_2 1 do 3 µm, Fluka Chemie AG, Švica in MnS 98 %, Alfa Johnson Matthey GmbH, Karlsruhe, Nemčija). Obliko in velikost delcev izbranih maziv smo preverili z laserskim granulometrom (Cilas Alcatel, Granulometer HR 850) in na vrstičnem elektronskem mikroskopu (SEM). Delež kisika v izdelanem Fe prahu in trdnih mazivih smo določili z analizatorjem kisika in dušika TC-436 Leco, ZDA. V mešalu turbula (Willy A. Bachofen AG Maschinenfabrik, Švica) smo nato s suhim mešanjem pripravili prašne mešanice z različnim deležem MoS_2 in MnS (0,5, 1,5 in 3 masnih odstotkov).

Sintezo kompozitov smo izvedli z vročim enoosnim stiskanjem prašnih mešanic v grafitnem orodju z uporabo visokofrekvenčnega (10 kHz) uporovnega ogrevanja. Takšen postopek se pri nas uporablja za pilotno proizvodnjo kompozitnih

not possible to measure the components of the cutting force at their origin, their reactions at some distance from the cutting point are recorded. Therefore we need a measurement system by which correct measurements can be performed independently of the action position of the force or moment. The goal of developing such a measurement system is to give to the industry simple and efficient measurement systems and therefore the possibility of applicative research. In Fig. 3, a measurement system for measurement the components of the cutting force for the turning processes is presented. The piezzo-electrical dynamometer transforms the mechanical signal into an electrical one, and this is further amplified with the charge amplifier. The frequency analyser, a required recording part of the chain, is needed because of the dynamical form of the force components and short cutting times of the cutting edge. The digitised time records are transferred and evaluated by the personal computer, where, with the suitable software equipment, the statical and dynamical values of the cutting force components are calculated.

3 SAMPLE PREPARATION AND MACHINABILITY TESTING

Practically pure technical iron powder was prepared by water atomisation [13]. After atomisation, the hardness of Fe particles was decreased by soft annealing in a laboratory tube furnace at 850° for three hours in dry hydrogen. During the atomisation, the chemical composition of the melt was controlled by the optical emission spectrometry (ARL, type 3460 Metals Analyzer, USA). Basic morphological and engineering properties (powder particle shape and size distribution, flowability, compressibility etc.) [18] of the prepared powder were also determined. The commercial chemically pure substances for laboratory use (MoS_2 1 to 3 µm, Fluka Chemie AG, Switzerland and MnS 98 %, Alfa Johnson Matthey GmbH, Karlsruhe, Germany) were used for the preparation of powder mixtures with different contents of MoS_2 and MnS. The particle size distribution and the shape of the selected lubricants were controlled by the laser granulometer (Cilas Alcatel, Granulometer HR 850) and by the scanning electron microscopy (SEM). Oxygen content of the prepared Fe powder and of the purchased lubricants was determined by the infrared light absorption based analysis (TC-436 Leco, USA). Powder mixtures with different contents of MoS_2 and MnS (0.5, 1.5 in 3 mass %) were finally prepared by dry mixing in a turbular blender (Willy A. Bachofen AG Maschinenfabrik, Switzerland).

The synthesis of composites was performed by uniaxial hot pressing of loosed powder mixtures in a multi cavity (5) graphite die coated with brusnih segmentov za krožne žage (diamantna zrna, vezana s kovinskim vezivom W-Cr-Cu) na polindustrijski stiskalnici (100 kN, dr. Fritch, Nemčija), ki omogoča relativno dobro regulacijo ter programirani nadzor vseh pomembnejših procesnih parametrov. Ustrezne zatehve (okoli 30 do 35 g) izbranih prašnih mešanic smo nasuli v poprej iz grafitnih ploščic sestavljeno petgnezdno orodje oziroma šablono. Prah smo še pokrili s ploščicami, ki so predstavljale zgornje pestiče. Tako sestavljeno in zaprto orodje je bilo primerno za neposredno namestitev na stiskalnico in vroče stiskanje.

Temperaturo smo merili z optičnim pirometrom na zunanji (črni; $\varepsilon \approx 1$) steni grafitnega orodja. Neposredno merjenje temperature s termoelementom v samem orodju ni mogoče zaradi prevelikih električnih motenj. Vroče stiskanje je potekalo v dveh fazah:

- predstiskanje do tlaka 35 MPa in hkratno ogrevanje orodja 5 min. do 960 °C,
- dokončno ogrevanje na 960 °C in stiskanje 10 min. z največjim tlakom (preklop na 45 MPa pri 900 °C).

Po izmetavanju je sledilo še hitro ohlajanje (5 do 6 min.; okoli 100 do 150 °C/min) vroče stisnjenih vzorcev na zraku. S preskusi smo ugotovili, da je bila 960 °C najvišja še sprejemljiva temperatura, tako v primeru kompozitov Fe-MoS_2 kakor tudi Fe-MnS. Pri daljših časih ali višjih temperaturah vročega stiskanja (970 do 980 °C) je prihajalo že do močnega lepljenja na orodje ali iztekanja taline. Pri nižjih temperaturah pa nismo dosegli zadovoljive zgostitve.

Na sedanji stiskalnici je mogoče tudi delo v zaščitni atmosferi, vendar smo menili, da njena uporaba v našem primeru ne bo potrebna, ker naj bi že samo grafitno orodje zagotavljalo dovolj reduktivno atmosfero [19].

Vroče stisnjenim vzorcem smo določili gostoto in trdoto ter iz njih izdelali metalografske obruse za preiskave na optičnem in mikroskopu SEM. Iz vzorcev smo izrezali tanke rezine in pripravili vzorce ($\approx\phi 3\times 5$mm), primerne za določitev deleža kisika, ogljika in žvepla. Na nekaterih tipičnih vzorcih so bile izvedene tudi mikroanalize EDS in AES. Nekaj vzorcev smo mehansko obdelali na dimenzije ϕ 8 × 40 mm, primerne za standardne tribološke preiskave (valjček na valju)([6] in [17]) in merjenje rezalnih sil.

Meritve rezalnih sil smo izvedli na eksperimentalni stružnici za fino vzdolžno struženje z oznako ES-2 (modifcirano za raziskave v Laboratoriju za odrezavanje, Fakultete za strojništvo, Ljubljana). Stružnica s svojimi lastnostmi zagotavlja nespremenljivost različnih vplivnih parametrov na velikost rezalnih sil tako,

boron nitride (BN). The die and powder mixtures were heated directly by the high frequency (10kHz) resistant heating system. Similar consolidation procedure is also used for the pilot production of composite segments for diamond-based grindstones on the semi-industrial hot press (100 kN, dr. Fritch, Germany) which enables relative good programming and control of all process parameters. The optimal hot pressing conditions of selected powder mixtures were determined experimentally by measuring dimensions and density of hot pressed samples.

The temperature was measured with an optical pyrometer on the black surface ($\varepsilon \approx 1$) of the graphite die, as close as possible to the hot pressing zone. Hot pressing was performed in two steps:

- pre-pressing up to 35 MPa and simultaneous heating of die/powder (5 min. up to 960°C),
- final heating and pressing (10′ at 960°C with maximal pressure 45 MPa).

After hot pressing and ejection, fast cooling of samples was performed (approximately 100 to 150°C/min.). It was established that 960°C is the maximal acceptable hot pressing temperature of the selected powder mixtures. Longer soaking times or higher temperatures of hot pressing causes reaction between Fe and the graphite die, powder melting and their sticking onto the die walls. However, at lower temperatures complete densification is not obtained.

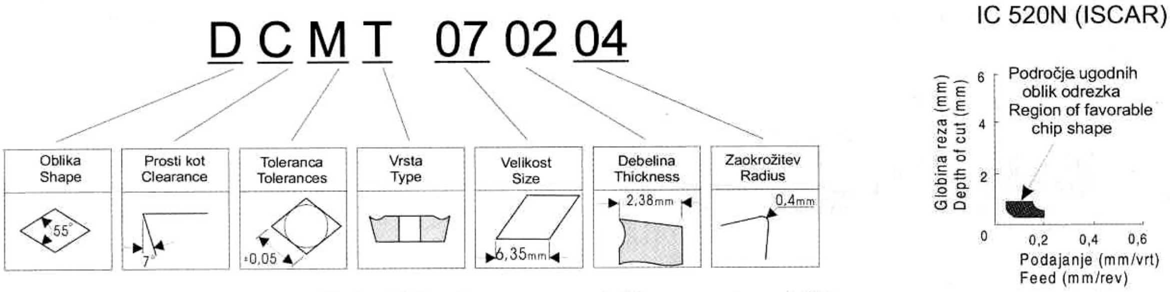
With this experimental procedure, almost completely densified samples with appropriate dimensions (10×10×40 mm) for metallographic, micro-analytical, mechanical and tribological investigations were prepared.

The existing experimental hot press also enables operation in a protective atmosphere. However, it was not used in our experimental work. The graphite die assures a reductive atmosphere which protects the composite against oxidation during hot pressing [19].

Density and hardness of hot pressed specimens were then determined. From metallographic samples, specimens were also prepared for investigations by the optical and scanning electron microscope, as well as EDS and AES microanalysis. Thin slices ($\approx\phi 3\times 5$mm) were cut off from the hot pressed specimens for the determination of oxygen, carbon and sulphur. Some specimens were machined into cylinders of appropriate dimensions (ϕ 8 × 40 mm) for tribological (pin on disc) ([6] and [17]) and machinability measurements.

The measurements of the cutting forces were performed on the experimental turning machine for the finish longitudinal turning with denotation ES 2 (modified for research work at the Laboratory for machining, Faculty of Mechanical Engineering, Ljubljana). The turning machine assure with its characteristics unchangeable differently influenced parameters which might influence da lahko iz izmerjenih rezultatov sklepamo o karakteristiki obdelovanih materialov. Preostali vplivni parametri - rezalno orodje iz oplaščene karbidne trdine, rezalna hitrost 100 m/min, podajanje 0,1 mm/vrt, globina reza 0,5 mm – se tudi niso spreminjali in so bili izbrani glede na priporočljive vrednosti za optimalno odrezovanje preskušanih materialov (sl. 4). Pred vsako meritvijo so bili obdelovanci centrirani, da ni prišlo do sprememb globine reza ali dodatnih vplivov na dinamične razmere v rezalni coni.

the magnitude of the cutting forces; therefore, from the measurement results, we can establish the characteristics of the workpiece materials. The other influence parameters - cutting tool from the coated carbide, cutting speed 100 m /min, feed 0.1 mm/rev, depth of cut 0.5 mm - also did not change, and have been selected according to the recomended values for the optimal cutting of tested materials (Fig. 4). Before each measurement the workpieces were centered in order to eliminate the changes in the depth of cut or additional influences on the dynamical circumstances at the cutting zone.



Sl. 4. *Oblika in sestava oplaščene rezalne ploščice*

Fig. 4. *The shape and composition of the coated carbide tool*

4 REZULTATI IN OBRAVNAVA

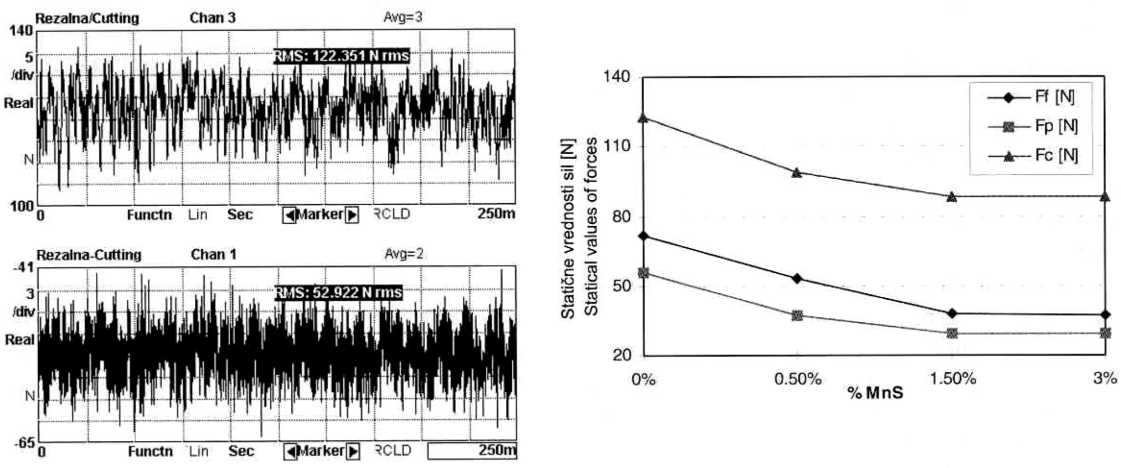
Na sliki 5 je prikazan izpis časovnega poteka rezalne sile pri struženju kompozita brez dodatka v primerjavi s kompozitom z dodatkom 0,5% MnS. Slika kaže tudi izmerjene vrednosti vseh treh komponent sil v odvisnosti od dodane količine dodanega MnS.

Iz zapisa statičnih vrednosti sil lahko ugotovimo, da obstajajo pomembne razlike v

4 RESULTS AND DISCUSSION

In Fig. 5 the time series record of the main cutting force in the turning of the composite without the added ingredient in comparison with the composite consisting 0.5 % MnS is shown. The figure also present the calculated statical values of all three forces in dependence on the MnS quantity supplemented.

From the calculated values of the statical forces we can establish that there exist significant differences in the

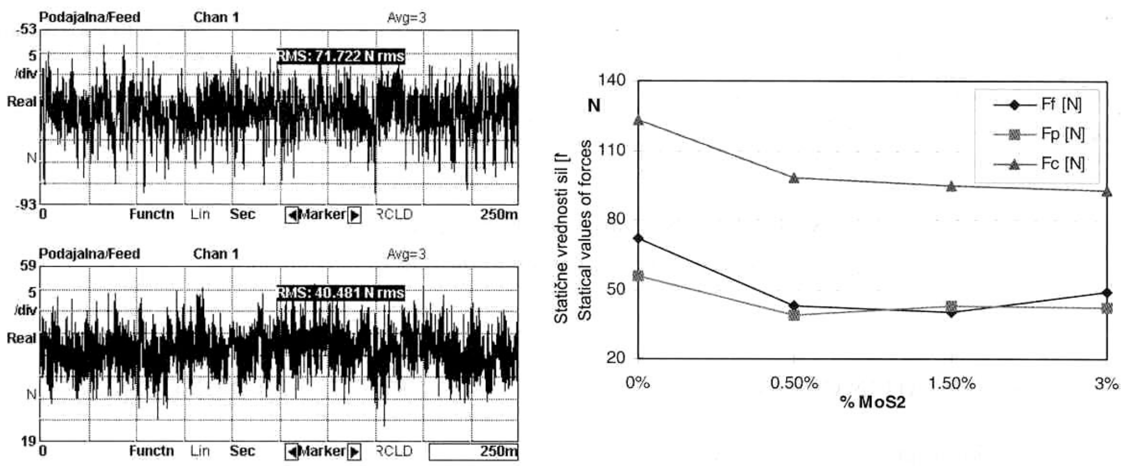


Sl. 5. *Izpis časovnega poteka rezalne sile in statične vrednosti komponent sil v odvisnosti od količine dodanega MnS*

Fig. 5. *Time record of the main cutting force and the values of the statical and dynamical force components in dependence on the MnS quantity supplemented*

velikosti sil pri struženju kompozita brez dodanega MnS in kompozita z 0,5 % ali 1,5 % dodatka MnS. Z večjo količino dodanega MnS se vrednosti sil ne zmanjšujejo. Iz tega lahko sklepamo, da se mazalne lastnosti kompozita z večanjem dodanega MnS čez mejo 1,5 % ne izboljšujejo. Vrednosti dinamičnih sil se z dodatkom MnS zmanjšujejo, kar pomeni, da dodatek MnS vsekakor vpliva na dušilne lastnosti kompozita. Vendar večanje količine dodatka MnS prek 0,5 % na dušenje bistveno ne vpliva. Podobne primerjave smo naredili tudi iz meritev sil na vzorcih z dodatkom MoS_2 (sl. 6).

force values in turning the composites without the MnS and composites with the supplement of the 0.5 % or 1.5 % MnS. However, with the higher quantity of the MnS supplemented, the values of the forces do not further decrease. From this we can establish that the lubrication ability of the composite does not improve above the limit of 1.5 % MnS supplemented. The values of the dynamical forces decrease with the MnS supplement, which means that MnS supplement certainly does influence the dumping characteristics of the composite. However the increase of the MnS supplemented quantity above 0.5 % does not significantly influence the dumping characteristics of the composite. Similar comparisons can be also be made from the measured results on the specimens with ingredients of the MoS_2. (Fig. 6).



Sl. 6. *Izpis časovnega poteka rezalne sile in statične vrednosti komponent sil v odvisnosti od količine dodanega MoS_2*

Fig. 6. *Time record of the main cutting force and values of the statical and dynamical force components in dependence on the MoS_2 quantity supplemented*

Iz velikosti izmerjenih statičnih sil lahko ugotovimo, da manjši dodatek MoS_2 (0,5%) samomazalno vpliva še bolj ugodno kakor MnS (izrazitejše zmanjšanje vrednosti sil). Z večanjem dodatka MoS_2 čez vrednost 0,5 % pa se sile bistveno ne spreminjajo. Po drugi strani pa manjši dodatek MoS_2 na velikost dinamičnih komponent sil ne vpliva značilno. Statistično ugotovljive vrednosti lahko ugotovimo šele pri dodatku 3 % MoS_2, kar pomeni, da le večja količina dodanega molibdenovega disulfida v kompozitu vpliva dušilno.

Izmerjene rezalne sile so dejanski odsev doseženih mikrostrukturnih in mehanskih lastnosti izdelanih kompozitov. Mikrostrukturne, kemijske in mikroanalitske preiskave so namreč pokazale, da je med sintezo kompozitov prišlo do delne oksidacije in odgorevanja dodanega maziva, še posebej med sintezo kompozita Fe-MoS_2 (preglednica1). Večji, ko je bil delež dodanega sulfida, večje je bilo v obeh primerih tudi odgorevanje S. V preglednici vidimo, da se s povečanim deležem sulfidnih delcev rahlo

From the calculated values of the statical forces we can establish that a lower quantity of MoS_2ingredient (0.5 %) influences the self-lubricating even more than the MnS (distinctive decrease of the force values). With an increase of the MoS_2 ingredient over the value of 0.5 % the forces do not change significantly. On the other hand, the lower quantity of the MoS_2 ingredient does not significantly influence the values of the dynamical forces. The statistical differences cannot be established until the ingredient of MoS_2 exceeds 3 %. This means that a higher quantity of the molibden disulphide incorporated in the composite can have a damping influence.

The determined cutting forces reflect the obtained microstructural and mechanical properties of the prepared composites. Microstructural, chemical and microanalytical investigations showed that during the synthesis of composites, partial oxidation and burning off of the added lubricant occurred. It is particularly evident for the Fe-MoS_2 composite (Table 1). In Table 1 it can be noticed that with the increased content of sulphur in powder mixture its burning-off increases. It can also be noticed that with increased content of solid lubricant hardness decreases and the densification ratio inmanjša trdota in veča stopnja zgostitve. Ta je izračunana na teoretično sestavo, ki pa v praksi ni dosežena. V resnici so stopnje zgostitve zato še višje. Iz metalografskih posnetkov ocenjujemo, da so dosežene gostote kompozitov nad 99,5% T.G. (teoretične gostote).

creases. Relative densities or densification ratio are calculated on the basis of theoretical chemical composition. The actual chemical compositions differ from the theoretical ones. Therefore, the obtained densification ratios are higher. Metallographic investigations show that the obtained densities of composites are above 99.5% of T.D. (theoretical density).

Preglednica 1. *Povprečne zgostitve, trdote in kemične analize vroče stisnjenih kompozitov*
Table 1. *Average densities, hardness and chemical analysis of hot pressed composites*

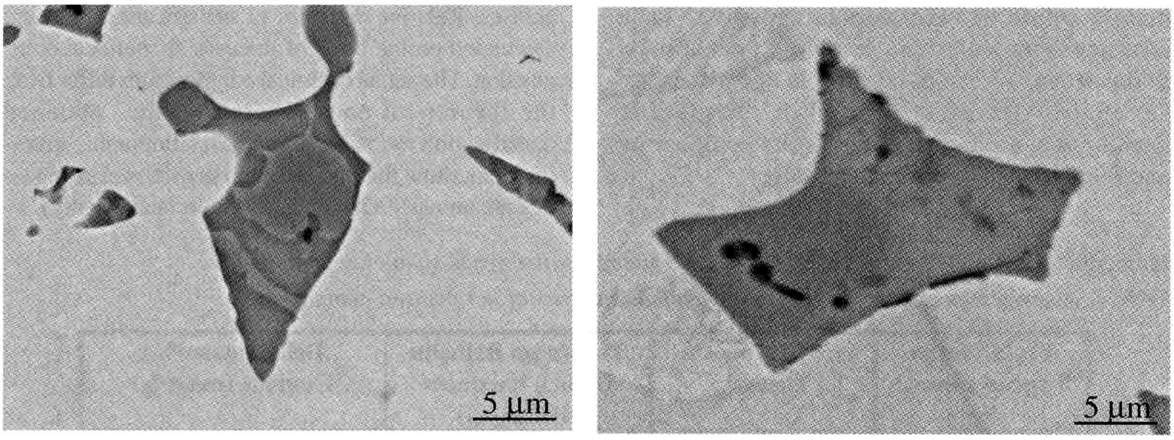
Vrsta vzorca Type of sample	**Gostota Density**		**Trdota po Brinellu Brinell hardness ($HB_{187,5/2,5}$)**	**Delež (masni%) Content (mass%)**		
	g/cm^3	**% T.G.**		**O**	**C**	**S**
Fe	7,68	97,6	159	0,46	0,029	0,02
Fe-0,5%MoS_2	7,71	98,5	183	0,21	0,033	0,22
Fe-1,5%MoS_2	7,67	98,6	175	0,47	0,028	0,48
Fe-3,0%MoS_2	7,71	100,0	155	0,49	0,021	0,60
Fe-0,5%MnS	7,63	97,6	164	0,64	0,013	0,21
Fe-1,5%MnS	7,61	97,8	159	0,69	0,021	0,53
Fe-3,0%MnS	7,53	97,8	156	0,68	0,027	0,84

Čeprav je MnS termodinamično bolj stabilen kakor FeS, je prišlo do njegove delne pretvorbe med vročim stiskanjem. Prišlo je tudi do delne oksidacije delcev MnS (sl. 7a) zaradi navzoče oksidativne atmosfere, med vročim stiskanjem. Vodno atomizirani delci Fe prahu so namreč površinsko oksidirani. Med hitrim vročim stiskanjem prahu obstaja možnost, da so med delci prahu ujete molekule zraka. Zanemariti ne smemo tudi tega, da je bil že sam izhodni MnS (enako MoS_2), uporabljen za pripravo mešanic, delno površinsko oksidiran. Kljub vsemu nam je z vročim stiskanjem uspelo izdelati kompozit Fe-(Mn,Fe)S z razmeroma dobro nadzorovanim deležem žvepla oziroma drobnih vključkov (Mn,Fe)S, ki so enakomerno razporejeni v kovinski matrici (sl. 1a), kar se kaže tudi na izmerjenih rezalnih silah.

Med vročim stiskanjem prašne mešanice Fe-MoS_2 so razmere drugačne. V navzočnosti molekularnega kisika MoS_2 zgoreva (MoS_2+$3O_2$=>MoO_2+$2SO_2$). Običajno se najprej tvori manj stabilni MoO_3 ([6] in [8]), ki je tekoč že nad 818 °C in hlapen nad 1155 °C. Čeprav ima Mo manjšo afiniteto do kisika kakor Mn, ima oksidacija bolj drastičen vpliv na sintezo Fe-MoS_2 kakor pa na sintezo kompozita Fe-MnS, saj je MoS_2 termodinamično manj stabilen od MnS. Pri temperaturah nad 1100°C je MoS_2 tudi termodinamično manj stabilen od FeS. Hkrati je Mo dobro topen v trdnem α- in γ-Fe. Zato so izpolnjeni vsi termodinamični pogoji za popolen razpad preostalega (neoksidiranega) MoS_2 in nastanek FeS (MoS_2+2Fe=>Mo+2FeS), ki je pri temperaturah sinteze tekoč (nad 988 °C pri tlaku 1 bar).

Although MnS is thermodynamically more stable than FeS, its partial decomposition occurs. The partial oxidation of MnS particles also occurred (Fig. 7a) during hot pressing because of the presence of oxidative atmosphere. The oxidative atmosphere is primarily a consequence of surface oxides formed during water atomization of Fe particles. During fast hot pressing of powder mixtures, molecules of adsorbed air can also be entrapped in the compact. The surface oxidation of added solid lubricants must not be neglected, either. In spite of that, a relatively good selflubricating Fe-(Mn,Fe)S composite with fine dispersion of (Mn,Fe)S particles is produced (Fig. 1a) which is reflected also in the measured cutting forces.

During the synthesis of the Fe-MoS_2 composite, the conditions are different compared with the synthesis of the Fe-MnS composite. In the presence of molecular oxygen, MoS_2 burns intensively (MoS_2+$3O_2$=>MoO_2+$2SO_2$). Usually less stable MoO_3 forms first ([6] and [8]). It is liquid already above 818°C and evaporates above 1155°C. Although Mo has lower affinity to oxygen than Mn, its oxidation has a more drastic influence on the synthesis of its Fe based composite. MoS_2 is also thermodynamically less stable than FeS at temperatures above 1100°C. Simultaneously, Mo has very good solid solubility in α- and γ-Fe. Therefore, all thermodynamic conditions are fulfilled for the full decomposition of the retained (nonoxidised) MoS_2 and for the formation of iron sulphide (MoS_2+2Fe=>Mo+2FeS), which is liquid at the temperatures of the synthesis (theoretically above 988°C at 1 bar pressure).



Sl. 7. *Posnetka SEM mikrostrukture kompozitov, dobljenih z vročim stiskanjem prašne mešanice a) Fe-MnS in b) Fe-MoS_2 z dobro vidno heterogeno sestavo sulfidnih delcev (temno sivo-oksidirana področja; črno-poroznost)*

Fig 7. *SEM micrograph of microstructure of composites prepared by the hot pressing of selected powder mixtures: a) Fe-MnS and b) Fe-MoS_2 with well visible heterogeneous structure of sulphide particles (dark grey - oxidised regions; black - porosity)*

Rezultati merjenja rezalnih sil kažejo, da ima nastali kompozit relativno dobro obdelovalnost in samomazalne lastnosti in da nanje vplivajo v kovinski matrici med sintezo nastali vključki FeS. Ugodne samomazalne lastnosti FeS so opazili tudi že nekateri drugi raziskovalci, ki so analizirali tribokemične reakcije na jeklenih površinah in obdelovalnost jekel, ki vsebujejo žveplo.

The results of cutting forces measurements show that synthesised composites have good machinability and therefore also self-lubricating properties. This is a result of the presence of FeS inclusions formed during synthesis. Attractive self-lubricating properties of FeS were already noticed during the analysis of tribo chemical reactions on sliding steel surfaces, as well as studying the machinability of steels with increased sulphur content.

5 SKLEPNE UGOTOVITVE

S postopkom vročega stiskanja smo iz izbranih prašnih mešanic izdelali samomazalni kompozit Fe-(Mn,Fe)S. Zaradi zelo slabe termodinamične stabilnosti MoS_2 med vročim stiskanjem prašne mešanice Fe-MoS_2 pa je nastal kompozit Fe[Mo]-FeS. V obeh primerih nam je uspelo dobiti praktično popolnoma zgoščene kompozite. Na popolno zgostitev je ugodno vplival predvsem nastanek tekoče faze (FeS in MnS), ki je med zgoščevanjem pod tlakom polnila prazne prostore med trdnimi delci Fe. Med vročim stiskanjem je prišlo do nadaljnje oksidacije navzočih sulfidnih delcev, čeprav naj bi po nekaterih literaturnih podatkih stiskanje v grafitnem orodju zagotavljalo pogoje, pri katerih naj ne bi prišlo do nadaljnje oksidacije. S postopkom vročega stiskanja je zato mogoče izdelati čiste in neoksidirane samomazalne kompozite vrste Fe-(Mn,Fe)S le, če uporabimo zaščitno atmosfero ali vakuum in čiste ter neoksidirane vhodne surovine. Z vročim stiskanjem prašne mešanice Fe-MoS_2 ni mogoče izdelati čistega kompozita Fe-MoS_2, ker pride do razpada MoS_2 in zato nastanka kompozita Fe[Mo]-FeS.

5 CONCLUSIONS

From the selected powder mixtures, selflubricating Fe-(Mn,Fe)S composites were prepared by hot pressing. However, during the consolidation of selected Fe-MoS_2 powder mixtures by hot pressing, the Fe[Mo]-FeS composite was formed. In both cases, a practically complete consolidated composite was prepared. The formation of liquid phase (FeS in MnS) has a beneficial influence on complete densification. Under pressure, the formed liquid phase filled voids (pores), which are placed among (during hot pressing) deformed solid Fe particles. Additional oxidation of sulphides occurred during the synthesis of composites although in some previous investigations it was stated that hot pressing in the graphite die assures conditions where no additional oxidation is expected. Our investigations show that a completely consolidated Fe-(Mn,Fe)S self-lubricating composite with fine dispersion of (Mn,Fe)S particles can be prepared by hot pressing but only in the protective atmosphere or in vacuum and only if very pure and nonoxidised raw materials are used. Pure Fe-MoS_2 cannot be prepared by the hot pressing procedure because of decomposition of MoS_2 and therefore a Fe[Mo]-FeS composite is formed.

Raziskave so pokazale, da lahko glede na rezultate meritev rezalnih sil pri odrezovanju ovrednotimo dejanske mikrostrukturne in z njimi povezane mehanske lastnosti kompozitov. To pa so tudi neposredni kazalniki uporabnih lastnosti, kakor sta na primer izboljšana samomazalnost in z njo povezana dobra obdelovalnost.

Our investigations also showed that the results of cutting forces measurement can be a relatively good indicator of real micro-structural, as well as mechanical characteristics of PM composites. Direct evaluation of engineering properties, such as improvement in self-lubricity and machinability, is possible.

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