J. Medved[1], M. Godec[2], I. Paulin[2], S. Kores[3], M. Vončina[1] (SLO)
[1] University of Ljubljana, Faculty of Natural Sciences and Engineering, Department for Materials and Metallurgy, Ljubljana, Slovenia
[2] Institut of Metals and Technology, Lepi pot 11, 1000 Ljubljana
[3] Talum Tovarna aluminija d. d. Kidričevo, Tovarniška cesta 10, 2325 Kidričevo, Slovenia

Kemijska, mehanska in toplotna obraba orodij ob stiku z aluminijevimi livnimi zlitinami

Chemical, Mechanical and Heat Wear of Tools in Cast Aluminium Alloys

Povzetek

Tlačno litje je eden od vodilnih livarskih postopkov v sodobni industriji. V primeru tlačnega litja je talina v stiku z orodjem, medtem ko pride do kemične interakcije med orodjem, izdelanim iz orodnega jekla, in talino. Poleg tega pride tudi do mehanske in toplotne obrabe orodij. Visoka produktivnost zahteva visoko odpornost na te dejavnike.

V raziskavi smo uporabili vzorce iz jekel UTOP MO1 in RAVNEX HD, da bi preverili njihovo odpornost proti obrabi v aluminijevih zlitinah Al99,7 in AlSi12. S tem namenom je bila izdelana laboratorijska naprava za testiranje dveh različnih orodnih jekel v dveh različnih aluminijevih zlitinah pri dveh različnih temperaturah 4 ure pri 75 vrtljajih na minuto. Posledica interakcije je rast reakcijske plasti, ki se tvori iz treh ali štirih plasti. Odpornost orodnega jekla UTOP MO1 je boljša v staljeni aluminijevi zlitini AlSi12 kot v Al99,7, medtem ko je obrabna orodnega jekla RAVNEX HD v aluminijevih zlitinah veliko slabša. Debelina interakcijskega sloja se poveča z naraščajočo temperaturo.

Ključne besede: interakcija orodno jeklo/staljeni aluminij, obrabna odpornost, interakcijska plast, intermetalne faze iz sistema Al-Fe

Summary

Pressure casting is one of the leading casting processes in the modern industry. In the case of pressure casting the melt is in contact with the tool, whereas the chemical interaction between the tools, made of hot-working tool steel, and the melt occur. In addition, mechanical and heat wear of the tools also occurs. High productivity requires high resistance to these factors.

For the experiment, samples from UTOPMO1 and RAVNEX HD were used to test their wear resistance in aluminium alloys Al99.7 and AlSi12. With this aim a laboratory device was designed to test two different tool steels in two different aluminium alloys at two different temperatures for 4 hours at 75 revolutions per minute. The result of the interaction is the growth of a reaction layer, which is formed from three or four layers. The wear resistance of UTOPMO1 tool steel is better in AlSi12 aluminium alloy than in Al99.7, whereas the wear resistance of RAVNEX HD tool steel is much worse in molten casting aluminium alloys. The thickness of the interaction layer increases with the rising temperature.

Keywords: interaction tool steel/molten aluminium, wear resistance, interaction layer, intermetallic phases from system Al-Fe

1 Uvod

V mnogih postopkih litja, zlasti pa pri tlačnem litju, je talina v stiku z orodjem. Med postopkom litja pride do kemične interakcije med orodjem, ki je izdelan iz jekla za delo v vtročem, in talino. Poleg tega pride tudi do mehanske in toplotne obrabe orodja. Za visoko produktivnost je potrebna velika odpornost na te dejavnike. Kemična interakcija se pojavi med orodnim jeklom in staljenim aluminijem, ki v skladu z binarnim faznim diagramom Fe-Al tvori intermetalne faze. Najpogostejši fazi sta Al_5Fe_2 in $Al_{13}Fe_4$ (Al_3Fe), ki tvorita reakcijsko plast. Da bi dosegli optimalne mehanske in fizikalne lastnosti orodja, je treba omejiti oziroma preprečiti tvorbo interakcijskega sloja, hkrati pa je potrebno poznati tudi mehanizme nastanka.

Jekla, uporabljena v naši raziskavi, so UTOP MO1 (H11) in RAVNEX HD (SIJ Metal Ravne), ki se pogosto uporabljajo v skupini orodnih jekel za delo v vročem, kjer je glavni legirni element krom. Ta orodna jekla imajo visoko stopnjo utrjevanja zaradi sorazmerno nizke austenitizacijske temperature, ki je približno 1020 °C, dobro odpornost proti oksidaciji, odpornost na toplotno utrujanje, odpornost proti eroziji v kontaktu s tekočim aluminijem itd.[1,2]. Trdota po utrjevanju znaša med 50 in 56 HRC, po kaljenju s sekundarnim kaljenjem pa lahko dosežemo podobno trdoto. V ta namen je jeklo legirano s kromom, molibdenom in vanadijem, ki s precipitacijo sekundarnih karbidov utrdijo matrico. Po kaljenju dosežemo optimalno kombinacijo trdote in duktilnosti. Jeklo UTOP MO1 se uporablja za orodja za vroče kovanje, orodja za litje, orodja za izsekovanje in za izdelavo nožev, RAVNEX HD pa se večinoma uporablja za orodja za tlačno litje[2].

Odpornost proti obrabi, ki močno vpliva na življenjsko dobo orodnega jekla v stiku

1 Introduction

In many casting processes, especially in high pressure die casting, the melt is in contact with the tool. During the casting process the chemical interaction between the tool, made of hot-working tool steel, and the melt occurs. In addition, mechanical and heat wear of the tools also occurs. For a stong productivity a high resistance to these factors is required. The chemical interaction occurs between tool steel and molten aluminium, which forms intermetallic phases in accordance with the binary phase diagram Fe-Al. The most common phases are Al_5Fe_2 and $Al_{13}Fe_4$ (Al_3Fe), which form a reaction layer. In order to achieve the optimal mechanical and physical properties of the tools, it is necessary to limit or to prevent the forming of interaction layers, whereas the mechanisms of origin have to be known.

The steels used in our study are UTOP MO1 (H11) and RAVNEX HD (SIJ Metal Ravne) and are commonly used in the group of hot-working tool steels, where the main alloying element is chromium. Investigated tool steels have a good hardenability from a relatively low austenitization temperature of about 1020 °C, a good resistance to oxidation, tempering resistance, resistance to erosion with liquid aluminium, etc.[1,2]. Hardness after hardening is between 50 and 56 HRC, and after tempering with secondary hardening a similar hardness can be achieved. For this purpose, the steel is alloyed with chromium, molybdenum and vanadium, which, by precipitating secondary carbides, harden the matrix. After tempering, an optimal combination of hardness and ductility is achieved. The steel UTOP MO1 is used for hot-forging tools, die casting tools, punching tools and for production of knives and RAVNEX HD is s tekočim aluminijem, je odvisna od treh dejavnikov:

1. mehanski: zaradi visoke hitrosti in temperature taline med litjem v forme iz orodnih jekel pride do erozije materiala s površine orodja. Litje aluminija povzroča izmenične mehanske napetosti, kar vodi do zmanjšanja mehanskih lastnosti in do razpada[3];

2. kemični: zaradi raztapljanja atomov železa in drugih zlitinskih elementov iz orodnega jekla pride v aluminiju do tvorbe intermetalnih faz na medfazni površini med orodnim jeklom in tekočim aluminijem. Nastale intermetalne faze imajo različne fizikalno-kemijske lastnosti, kot pa osnova[4];

3. toplotni: toplotna utrujenost nastane zaradi porabe in krčenja jekla med delovnimi cikli pri litju aluminija. Orodje je običajno segreto na 400 °C, temperatura pa se lahko dvigne tudi do 700 °C. V tem primeru se površinske plasti jekla razširijo, kar je v nasprotju z notranjostjo jekla, posledično se na površini pojavijo tlačne, v jedru pa natezne napetosti. Ko odstranimo ulitek iz orodja, orodje mažemo z ločilnim sredstvom, ki orodje močno ohladi, kar povzroči natezne napetosti na površini in tlačne napetosti znotraj orodja. Te napetosti so zelo visoke in so blizu končne trdnosti orodnega jekla za delo v vročem. Ker je uporaba orodja ciklična in se ta postopek ponovi več kot deset tisočkrat, se na orodju pojavijo razpoke[1].

Na mejnih plasteh nastanejo intermetalne faze jekla/staljenega aluminija kot posledica kemijske reakcije orodnega jekla in aluminijeve taline. Pogoj za nastanek faz je optimalno omočenje in difuzija, ki izhaja iz razlike med kemijskimi potenciali elementov v tekočem aluminiju in trdnem orodnem jeklu. Na mejni fazi med trdnimi jeklenimi in intermetalnimi fazami, atomi aluminija in železa reagirajo in tvorijo nove intermetalne faze z uporabo atomov

mostly used for tools for high pressure die casting[2].

The wear resistance, that strongly affects the life of the tool steel in contact with liquid aluminium, depends on three factors:

1. Mechanical: due to the high speed and temperature of the melt during casting into permanent moulds from tool steels, erosion of material from the surface of the tool occurs. Aluminium die- casting produces alternating mechanical stresses, which leads to a reduction in mechanical properties and decay[3].

2. Chemical: due to the dissolution of iron atoms and other alloying elements from the tool steel in aluminium, the formation of intermetallic phases on the interphase surface between tool steel and liquid aluminium occurs. The resulting intermetallic phases have different physicochemical properties as the basis[4].

3. Heat: thermal fatigue occurs due to expending and shrinking of steel during working cycles at aluminium die-casting. The tool is usually preheated to 400 °C, but the temperature can rise to 700 °C. In this case, the surface layers of steel are expanded, which is in contrary to the interior of the steel, consequently on the surface the compression and at the core the tensile stresses appear. When the casting is removed from the tool, the tool is lubricated with a separating agent, which greatly cools the tool, causing tensile stresses on the surface and compression stresses inside the tool. These tensions are very high, close to the ultimate strength of the hot-working tool steel. Since cyclic use of the tool, this process repeats more than ten thousand times, cracks on the tool occur[1].

At the boundary layer tool steel/molten aluminium intermetallic phases as a result of the chemical reaction of the tool steel and aluminium melt are generated. The trdnih kovin, kar povzroča premikanje fazne meje v smeri orodnega jekla[5]. Poleg atomov železa in aluminija so v mejni (reakcijski) plasti prisotni tudi drugi zlitinski elementi, zlasti legirani elementi orodnega jekla, kot so silicij, mangan, krom, molibden in vanadij. Ti elementi na splošno zmanjšujejo debelino intermetalne plasti, največji učinek pa ima silicij. Rast intermetalne faze Al_5Fe_2 poteka prednostno v smeri [001] z difuzijo skozi prosta mesta in ima zato visoko orientirano morfologijo v obliki jezika. Raziskave[6,7,8] so pokazale, da je v fazi Al_5Fe_2 v tej smeri delež prostih mest 30 %. Domneva se, da atomi silicija zasedajo mesta v intermetalni fazi Al_5Fe_2, kar povzroča izkrivljanje kristalne rešetke in zmanjšanje aktivacijske energije. Da bi zmanjšali vsebnost prostih mest v Al_5Fe_2, se zmanjša tudi difuzijski koeficient železa in aluminija v fazi Al_5Fe_2 in posledično kinetika tvorbe intermetalnih faz. Posledično se spremeni morfologija faze Al_5Fe_2, znotraj te faze pa se tvorijo drobni delci $τ_1/τ_9$, značilni za trojni sistem Al-Fe-Si[9]. Selverian et al. [10] so odkrili, da dodajanje silicija v talino aluminija in cinka močno zmanjša eksotermičnost reakcije med aluminijem in jeklom, na katero se nanaša mazivo, saj silicij tvori trdno reakcijsko plast, ki deluje kot pregrada med jeklom in talino. Pregrada močno omejuje difuzijo atomov aluminija in železa, tako da se reakcijska kinetika močno zmanjša.

V študiji prednostne rasti Fe-Al intermetalnih faz v temperaturnem območju med 600–1050 °C, s poudarkom na tvorbi faz Fe_3Al in FeAl, so ugotovili, da se ti dve fazi pojavita le pri temperaturah nad 1000 °C. Faza Fe_3Al in FeAl imata večjo vsebnost železa, zato je njihova obrabna odpornost boljša. Nasprotno pa je dokazano, da se faze $FeAl_2$, Fe_2Al_5, in $FeAl_3$ tvorijo pri temperaturah pod 1000 °C in imajo višjo

condition for the formation of phases is the optimal wetting and diffusion, resulting from the difference between the chemical potentials of the elements in liquid aluminium and solid tool steel. At the phase boundary between solid steel and intermetallic phases, atoms of aluminium and iron react and form new intermetallic phases, using solid metal atoms, causing the movement of phase boundary in the direction of tool steel[5]. In addition to the atoms of iron and aluminium, other alloy elements, in particular the alloying elements of the tool steel, such as silicon, manganese, chromium, molybdenum and vanadium, are also present in the boundary (reaction) layer. In general, these elements reduce the thickness of the intermetallic layer, whereas the greatest effect has silicon. The growth of the intermetallic phase Al_5Fe_2 takes place preferably in the direction [001] by diffusion through the vacancies, and therefore have a highly oriented morphology in the form of a tongue. Researches[6,7,8] showed that the proportion of vacancies in this direction for the phase Al_5Fe_2 is 30 %. It is assumed that silicon atoms occupy gaps in the intermetallic phase Al_5Fe_2, causing the distortion of the crystal lattice and the reduction of activation energy. In order to reduce the vacancies content in Al_5Fe_2, the diffusion coefficient of iron and aluminium in the phase Al_5Fe_2 and, consequently, the kinetics of the formation of intermetallic phases, is also reduced. As a consequence, the morphology of the Al_5Fe_2 phase changes, and inside this phase fine particles $τ_1/τ_9$, characteristic for the ternary Al-Fe-Si system, are formed[9]. Selverian et al.[10] have discovered, that the addition of silicon to the melt of aluminium and zinc greatly reduces the exothermic nature of the reaction between aluminium and steel on which the lubricant is applied, since silicon forms a solid reaction layer acting as a barrier between steel and melt.

vsebnost aluminija. Te faze so krhke in zato manj obstojne[11,12].

Pri preučevanju interakcijskega sloja med legiranim jeklom H13 v talini zlitine AlSi9Cu3 in pri kontaktnem času 500 s je bilo dokazano, da debelina intermetalne plasti hitreje raste pri višjih temperaturah in istem kontaktnem času[13]. S časom se debelina intermetalnih plasti poveča, hitrosti rasti intermetalnih faz pa se spreminjajo. Dokazano je, da rast faze Fe_2Al_5 poteka po paraboličnem zakonu, kar na začetku (v kratkem času testiranja) ne velja. Rast fazne meje na podlagi $FeAl_3$ faze poteka linearno glede na čas[14].

S tem ciljem je bil preučen vpliv medsebojnega delovanja dveh različnih aluminijevih zlitin in orodnih jekel za delo v vročem. Preučena je bila obraba vzorcev vroče obdelanih orodnih jekel UTOP MO1 in RAVNEX HD in reakcijska plast, ki je bila vzpostavljena na meji med tekočim aluminijem in jeklom, kot rezultat kemijske reakcije. Poskusi so bili izvedeni na posebej zasnovani napravi, medtem ko so bili vzorci metalografsko analizirani, z namenom določitve interakcije.

2 Eksperimentalno delo

Za eksperimentalne vzorce so bili uporabljeni vzorci iz orodnega jekla, katerih kemična sestava je prikazana v tabeli 1.

Shema uporabljenega vzorca je prikazana na sliki 1a. Poskusi so bili izvedeni na laboratorijski napravi, prikazani na sliki 1b. Napravo sestavljajo talilna peč z električnim uporom s krmilnim sistemom, s katerim se vzdržuje konstantna temperatura; nosilec, na katerega je bil nameščen elektromotor in keramični lonček za talino. Eksperimentalni vzorec je pritrjen na elektromotor s pomočjo jeklene ali

The barrier greatly limits the diffusion of the atoms of aluminium and iron, so that the reaction kinetics is greatly reduced.

In the study of the preferred growth of Fe-Al intermetallic phases in the temperature range between 600–1050 °C, with the emphasis on the formation of Fe_3Al and FeAl phases, it was found that these two phases occur only at temperatures above 1000 °C. Fe_3Al and FeAl phases have a higher iron content, and therefore their wear resistance is better. Conversely, it has been shown that the $FeAl_2$, Fe_2Al_5, and $FeAl_3$ phases are formed at temperatures below 1000 °C and have a higher aluminum content. These phases are fragile and therefore less persistent[11,12].

In the study of the interaction layer between the H13 alloy steel in the alloy melt AlSi9Cu3 and at the contact time of 500 s, it was demonstrated that at higher temperatures and the same experiment time, the thickness of the intermetallic phase layer is growing faster[13]. With time, the thickness of the intermetallic phases in layer increases, whereas the growth rates of the intermetallic phases varies. It has been proven that the growth of the Fe_2Al_5 phase is carried out according to the parabolic law, which initially (at short testing time) does not apply to this law. The growth of the phase boundary on the basis of the $FeAl_3$ phase takes place linearly with respect to time[14].

With this aim, the influence of the interaction between two different aluminium alloys and hot- working tool steels was studied. The wear of the sample from the UTOP MO1 and RAVNEX HD and the reaction layer, which is established at the boundary between liquid aluminium and steel as a result of a chemical reaction, was investigated. Experiments were performed on a specially designed device, whereas the samples were metallographically analysed in order to determine the interaction.

Tabela 1. Kemijska sestava jekel UTOP MO1 in RAVNEX HD v masnih odstotkih v skladu z internim standardom SIJ Metal Ravne[2]

Table 1. Chemical composition of UTOP MO1 and RAVNEX HD in wt. % according to the internal standard SIJ Metal Ravne[2]

	C	Si	Mn	Cr	Mo	V	Ni
UTOP MO1	0,37	1,00	0,38	5,15	1,30	0,40	-
RAVNEX HD	0,36	0,30	0,40	5,00	1,7	0,60	1,65

grafitne palice, ki vrti vzorec s vrtenjem (vrt./min) v talini aluminijeve zlitine.

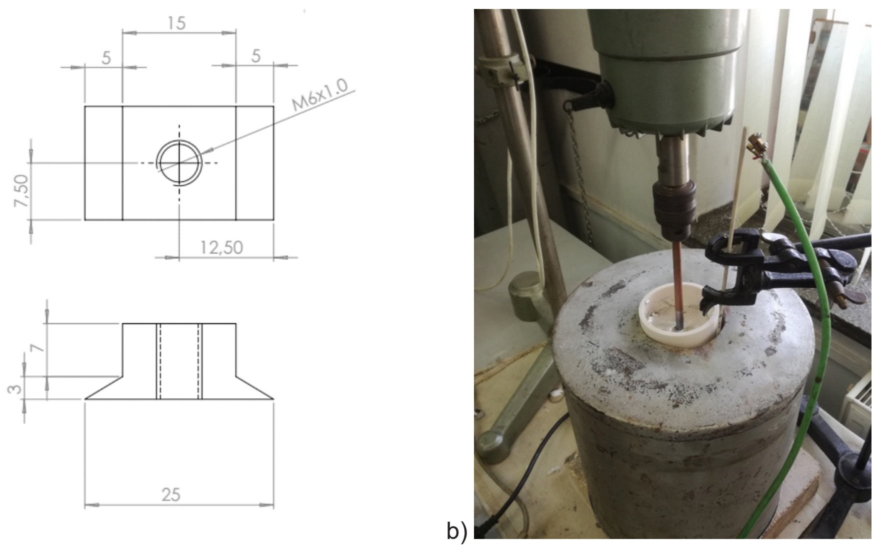
Najprej je bila Al-zlitina staljena v električni uporni peči. Vhodni parametri, uporabljeni med poskusi, so prikazani v tabeli 2. Ko je temperatura taline dosegla 670 ali 700 °C, se je vzorec dalo v talino, pri čemer je bila naprava za vrtenje vzorca vklopljena 4 ure pri 70 ali 150 vrt./min. Po končanem poskusu so bili vzorci vzeti iz peči in ohlajeni na zraku.

Za metalografsko analizo reakcijske plasti med jeklom in Al-zlitino so bili vzorci

2 Experimental Work

For the experiment samples from the tool steel were used, which chemical composition is shown in Table 1.

The scheme of the used sample is shown in Fig. 1a. The experiments were carried out on the laboratory device shown in Fig. 1b. The device consists of an electric resistant melting furnace with a control system with which a constant temperature was maintained, the carrier on which the electric motor was placed and the ceramic



a) b)

Sl. 1. Shematski prikaz vzorca orodnega jekla (a) in laboratorijske naprave za določanje interakcije med orodnim jeklom in tekočim aluminijem (b)

Fig. 1. Schematic representation of the tool steel sample (a) and laboratory device for determining the interaction between tool steel and liquid aluminium (b)

Tabela 2. Uporabljeni materiali in parametri za izvedbo poskusov

Table 2. Used materials and parameters to perform the experiments

Vzorec / Sample	Jeklo / Steel	Talina / Melt	Temperatura taline / Melt temperature [°C]	Rpm [min^{-1}]	Čas / Time [h]
1	RAVNEX HD	Al99,7	670	70	4
2	RAVNEX HD	Al99,7	700	150	4
3	UTOP MO1	Al99,7	670	150	4
4	UTOP MO1	Al99,7	700	150	24
5	UTOP MO1	AlSi12	600	70	4
6	UTOP MO1	AlSi12	700	70	4

prerezani na pol. Vzorci so bili pripravljeni po standardnem metalografskem postopku. Na mikroskopu Olympus BX 61 je bila izvedena svetlobna mikroskopija. Identifikacija faz in interakcijskih plasti v pregledanih vzorcih je bila izvedena na elektronskem mikroskopu (SEM) JEOL JSM-5610 na Naravoslovnotehniški fakulteti. Del SEM analiz pa je bilo opravljenih tudi na Inštitutu za kovinske materiale in tehnologije s SEM JSM-6500F.

3 Rezultati in diskusija

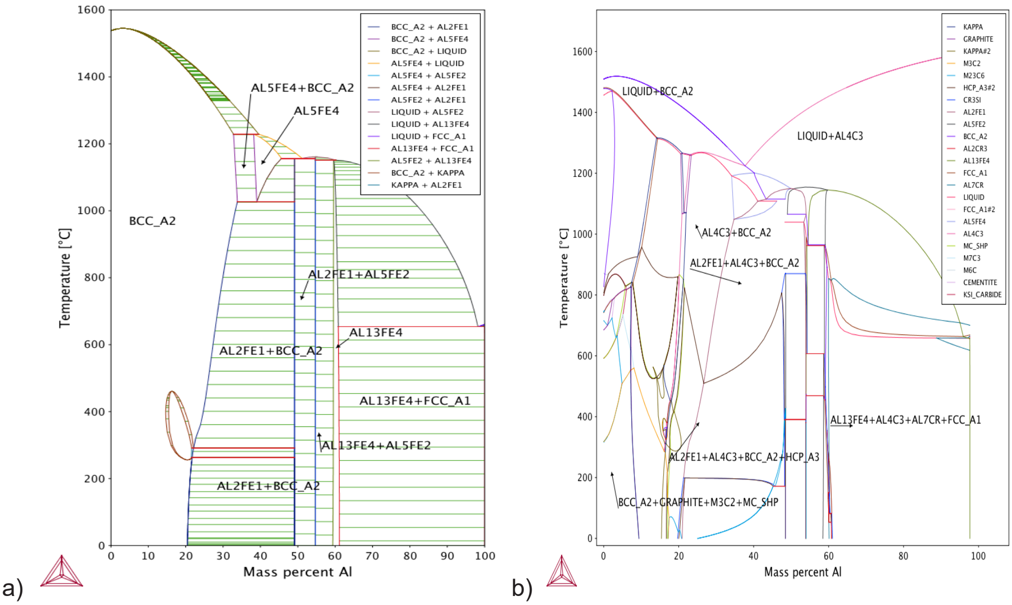
Interakcija med orodnim jeklom in staljenim aluminijem je bila termodinamično proučena z modeliranjem faznih ravnotežij in faznih diagramov. V sistemu Al-Fe so prisotne tri faze $Al_{13}Fe_4$, Al_5Fe_2 in Al_2Fe (slika 2a). V sistemu Al-Fe-C-Cr-Mo pa so poleg omenjenih binarnih faz prisotni tudi karbidi, pri čemer se lahko tvori tudi aluminijev karbid. Aluminij s kromom tvori fazo Al_7Cr, molibden pa Mo_3Al fazo (slika 2b).

Slika 3 prikazuje makro slike eksperimentalnih vzorcev jekla v zlitini Al99,7 in AlSi12. Vidna je oblika orodnega jekla, obdana z aluminijem. Že iz makro posnetkov se vidi reakcijsko območje med jeklom in aluminijem, pa tudi razpoke in poroznost zaradi krčenja med strjevanjem aluminija. Obraba vzorcev je različna in je

crucible for the melt. The experimental sample is fixed to an electric motor via a steel or graphite rod, which rotates the sample with a constant revolution per minute (rpm) in aluminium alloy melt.

Firstly, the Al-alloy was melted in an electric resistance furnace. The input parameters, which were used during the experiments, are shown in Table 2. When the temperature of the melt reached 600, 670 or 700 °C the sample was placed into the melt and the device for rotating the sample was turned on for 4 hours at 70 or 150 rpm. After the experiment was completed, the samples were taken from the furnace and left to cool in air.

For the metallographic analysis of the reaction layer between the steel and the Al-alloy, the samples were cut in half. The samples were prepared by standard metallographic procedure. Light microscopy was performed on the Olympus BX 61 microscope. The identification of phases and interaction layers in the examined samples was carried out on the scanning electron microscope (SEM) JEOL JSM-5610 at the Faculty of Natural Sciences and Engineering and JEOL JSM-6500F at the Institute for Metals, Materials and Technologies.



Sl. 2. Fazna ravnotežja v sistemih Al-Fe (a) in Al-Fe-C-Cr-Mo (b)

Fig. 2. Phase equilibria in Al-Fe (a) and Al-Fe-C-Cr-Mo (b) systems

odvisna od temperature ter časa preskusa, kar je razvidno iz geometrije vzorca. Na obrabo ima večji vpliv število obratov kot temperatura. Največja obraba je razvidna iz vzorca 4, kjer je preskus trajal 24 ur.

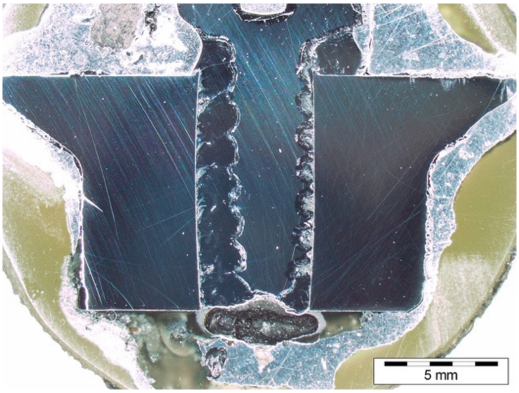
Vsi vzorci so bili analizirani po istem postopku. Mikrostrukture reakcijskih plasti med orodnim jeklom in Al99,7, odvzete na istem mestu (vrh in rob) vzorcev, so prikazane na sliki 4. Plast je sestavljena iz dveh območij. Na aluminijasti strani je plast najverjetneje sestavljena iz Al-Fe in Al-faze. Na jekleni strani pa je plast v obliki prstov, ki rastejo v jeklo. Pri aluminiju opazimo povečan delež železnih faz (igel).

Iz mikrostrukture vzorcev 1 in 2 je razvidno, da je vmesni reakcijski sloj pri vzorcu, ki je bil preizkušan pri višjih vrtljajih, porozen. Reakcijska cona je pri nižjih vrtljajih debelejša. Vzorca 2 in 4 smo preskusili pri višji temperaturi. Iz

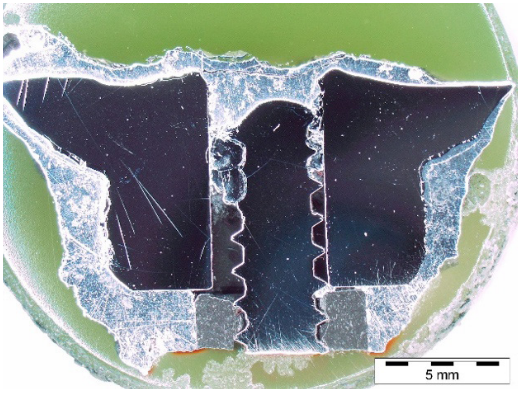
3 Results and Discussion

The interaction between tool steel and molten aluminium was studied thermodynamically by modelling phase equilibria and phase diagrams. There are three phases in the Al-Fe system: $Al_{13}Fe_4$, Al_5Fe_2 and Al_2Fe (Fig. 2a). In the Al-Fe-C-Cr-Mo system, besides the mentioned binary phases, there are also carbides, whereas the aluminium carbide can also be formed. Aluminium with chromium forms the Al_7Cr phase, and with molybdenum Mo_3Al phase (Fig.2b).

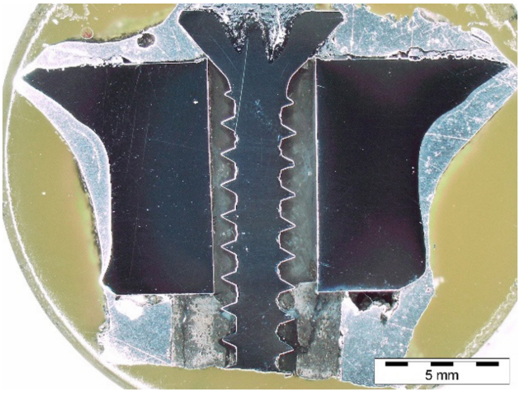
Fig. 3 shows macro images of tested steel specimens in alloy Al99.7 and AlSi12. The patterns of tool steel, surrounded by aluminium, are seen. Already from the macro shots, the reaction zone between steel and aluminium is seen, as well as cracks and porosity due to shrinkage during the hardening of aluminium. The wear of



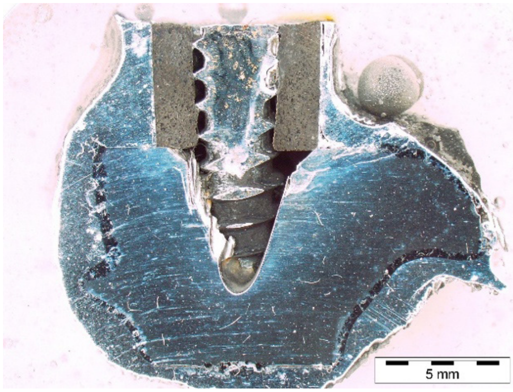
1: RAVNEX HD, Al99.7, 670 ºC, 70 min^{-1}, 4h



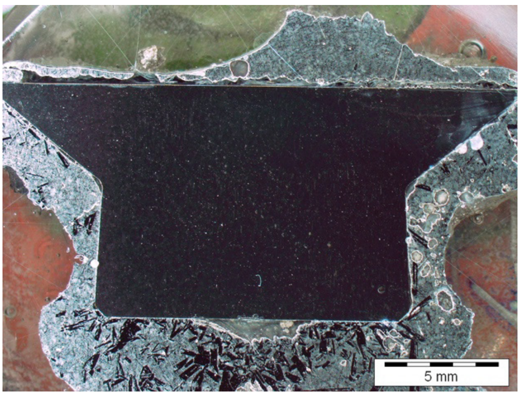
2: RAVNEX HD, Al99.7, 670 ºC, 150 min^{-1}, 4h



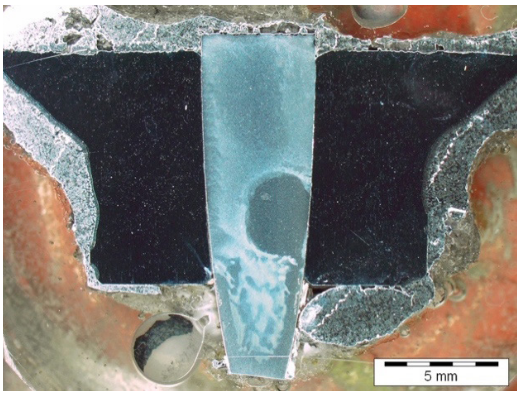
3: UTOP MO1, Al99.7, 670 ºC, 150 min^{-1}, 4h



4: UTOP MO1, Al99.7, 700 ºC, 150 min^{-1}, 24h



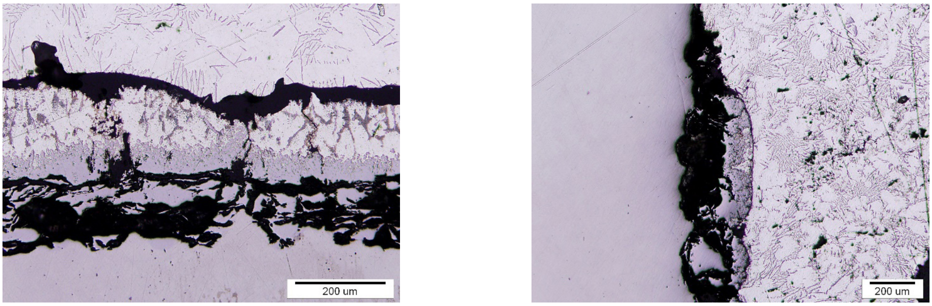
5: UTOP MO1, AlSi12, 670 ºC, 70 min^{-1}, 4h



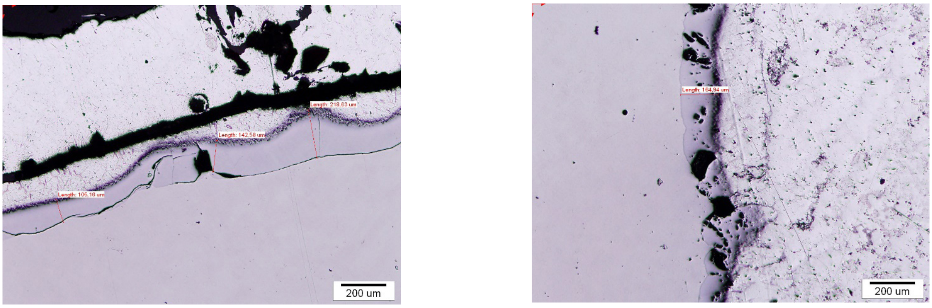
6: UTOP MO1, AlSi12, 700 ºC, 70 min^{-1}, 4h

Sl. 3. Makro posnetki preiskovanih vzorcev

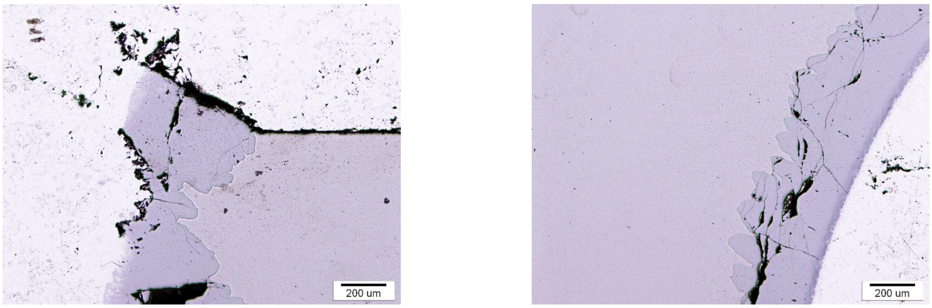
Fig. 3. Macro image of investigated samples



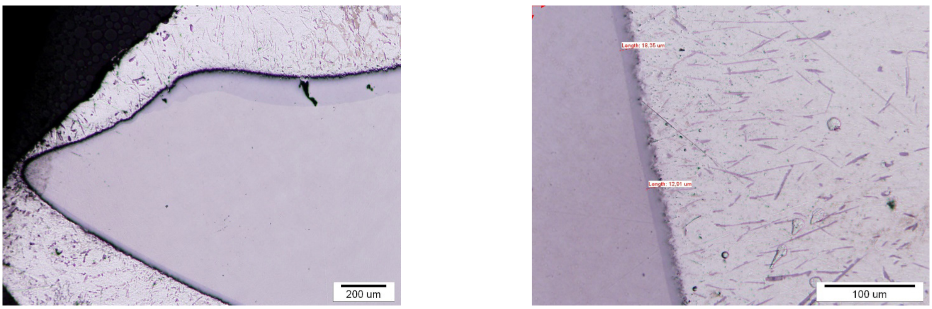
1: RAVNEX HD, Al99,7, 670 ºC, 70 min^{-1}, 4 h



2: RAVNEX HD, Al99,7, 670 ºC, 150 min^{-1}, 4 h



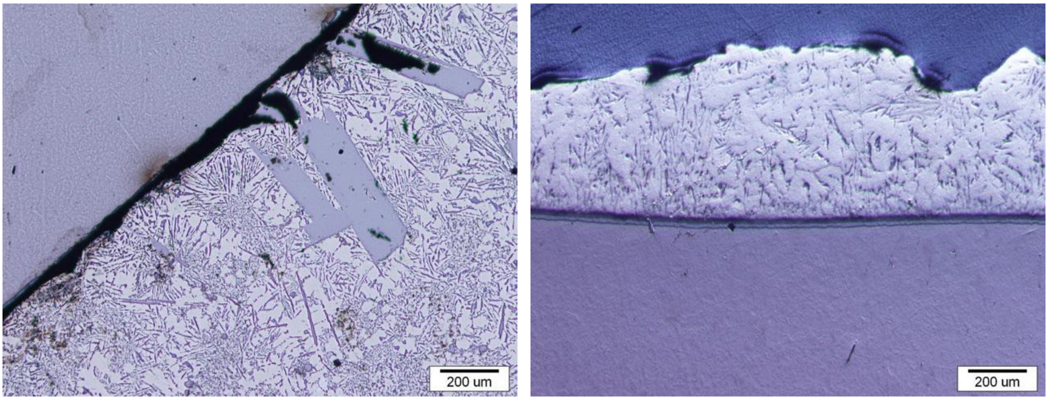
3: UTOP MO1, Al99,7, 670 ºC, 150 min^{-1}, 4 h



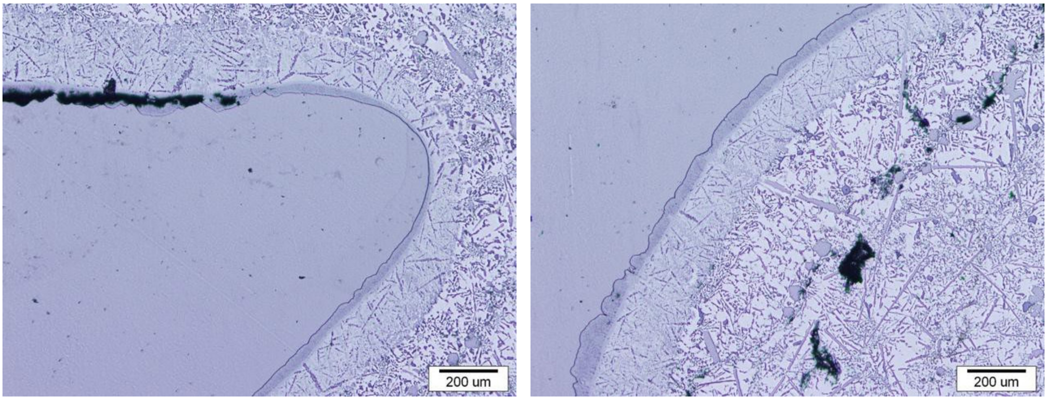
4: UTOP MO1, Al99,7, 700 ºC, 150 min^{-1}, 24 h

Sl. 4. Mikrostrukture reakcijskega sloja orodnega jekla/Al99,7

Fig. 4. Microstructures of the reaction layers tool steel / Al99.7



5: UTOP MO1, AlSi12, 600 ºC, 70 min^{-1}, 4h



6: UTOP MO1, AlSi12, 700 ºC, 70 min^{-1}, 4h

Sl. 5. Mikrostrukture reakcijskega jekla orodje jekla/AlSi12

Fig. 5. Microstructures of the reaction layers tool steel / AlSi12

mikrostrukture na sliki 4 je mogoče videti, da je reakcijska plast debelejša in ima večjo poroznost. V aluminiju se pojavijo razpoke takoj za plastjo s povečano železovo fazo v obliki igel.

Reakcijski sloj je razpokan, razpoke pa so vidne tudi v aluminiju, kjer opazimo obsežna območja evtektike (največ raztopljenega železa). Po razpokah reakcijske plasti se reakcijski sloji razgradijo in pomešajo s tekočim aluminijem. Takšne faze potrebujejo veliko časa, da se raztopijo v aluminiju. Odpornost jekla UTOP MO1 je the samples is different and depends on the temperature and the time of the test, as can be seen from the geometry of the sample. The wear is more influenced by rpm than the temperature. The highest wear can be seen from sample 4, where the experiment lasted for 24 h.

All samples were analysed by the same procedure. Microstructures of reaction layers between tool steel and Al99.7 taken in the same place (top and edge) of the samples are shown in Fig. 4. The layer is composed of two sections. On the višja v primerjavi z jeklom RAVNEX HD zaradi tanjše reakcijske plasti in manjšega deleža razpok.

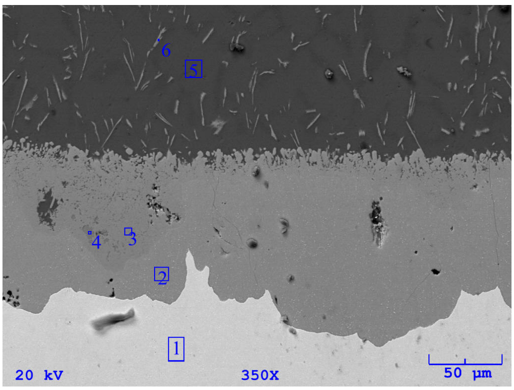
Za primerjavo je bila preizkušena odpornost proti obrabi UTOP MO1 v aluminijevi zlitini AlSi12 pri temperaturi 600 in 670 °C (slika 5). Reakcijska plast vzorca 5 (nižja temperatura) je oblikovana iz enakomerne plasti. Veliko debelejši reakcijski sloj je viden pri vzorcu 6 (višja temperatura), ki je sestavljen vsaj iz treh različnih slojev. Ocenjeno je bilo, da se delež železa v aluminiju blizu fazne meje poveča zaradi boljše difuzije železa, medtem ko vsaj ena plast vsebuje tudi večji delež silicija.

Na sliki 6, na kateri je prikazan SEM posnetek z EDS analizo vzorca 1, opazimo v staljenem aluminiju rast intermetalnih Fe-faz v obliki jezika. Razlikujemo dve različni plasti, ena je kompaktna, nekoliko svetlejša in debelejša (bližje jeklu), druga pa v obliki aluminium side, most likely it consists of Al-Fe and Al-phase. On the steel side there is a layer in the form of fingers that grow into steel. An increased proportion of iron phases (needles) is observed in aluminium.

From the microstructure of the samples 1 and 2, the intermediate reaction layer is porous in sample tasted at higher rpm. The reaction zone at lower rpm is thicker. Samples 2 and 4 were tested at a higher temperature. From the microstructure in Fig. 4 it can be concluded that the reaction layer is thicker, with more porosity. In the aluminium, cracks appear immediately behind the layer with an increased Fe-based needle phase.

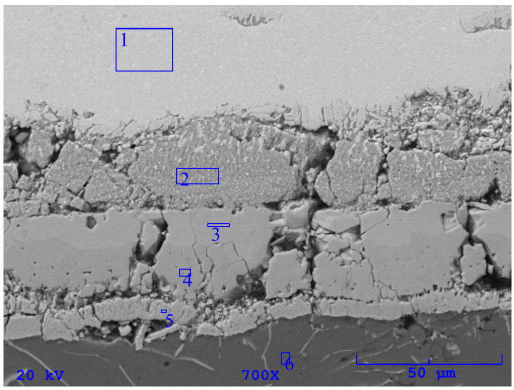
The reaction layer is cracked, and cracks are also visible in aluminium, where extensive areas of eutectics (most dissolved iron) are observed. Such phases need a lot of time to dissolve in aluminium. The resistance of steel UTOPMO1 is higher

(a)



1 (mas.%): Cr 4,5, Mo 1,8, V 0,71, Fe ostalo
2 (mas.%): Fe 45, Cr 2,7, Mo 0,73, Al ostalo
3 (mas.%): Fe 37, Cr 2,2, Mo 0,72, Al ostalo
5 (mas.%): Al 100
6 (mas.%): Al 91, Fe 9

(b)



1 (mas.%): Cr 4,7, Mo 1.8, V 0,6, Fe ostalo
2 (mas.%): Cr 10,2, Mo 4,1, V 1,3, Si 0,9, Fe ostalo
3 (mas.%): Cr 0,3, O 4,6, Fe ostalo
5 (mas.%): O 5,6, Fe ostalo
6 (mas.%): Fe 0,5, Al ostalo

Sl. 6. Mikroposnetki in EDS analiza interakcijskega sloja v vzorcu 1 (a) in 4 (b)

Fig. 6. Micrograph and microanalysis of the interaction layer in the sample 1 (a) and 4 (b)

igel ali jezika. EDS analize predstavljajo: 1 – sestava jekla, 2 - faza Al-Fe v stiku z orodnim jeklom z večjim deležem kroma, 3 in 4 - faza Al-Fe z manjšo količino železa bližje aluminiju, 5 - sestava aluminija in 6 – Fe-faza iz aluminija. Koncentracija aluminija je večja v območju plasti, ki je bližje aluminijevi zlitini, ki je lahko faza $Al_{13}Fe_4$ in se zmanjša, ko je bližje jeklu, kar bi lahko bila faza Al_5Fe_2. Plast bližje aluminiju je razdrobljena, pri čemer se železo raztopi v staljenem aluminiju v obliki igel iz Fe-faze.

Slika 6. predstavlja mikrostrukturo in mikroanalizo vzorca 5 in 6, kjer je orodje jeklo UTOP MO1 v stiku z zlitino AlSi12.

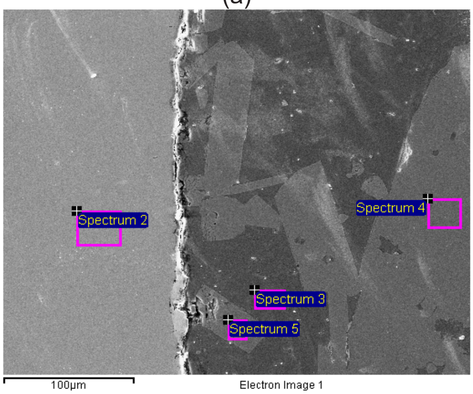
V vzorcu 5 (slika 7 (a)) je reakcijska plast tanka z široko plastjo raztopljenih Fe-faz v zlitini AlSi12. Pri višji temperaturi je obraba vzorca veliko večja, reakcijski sloj je sestavljen iz treh plasti, kar je razvidno iz mikrostrukture in mikroanalize EDS. Večje Fe-faze plavajo v aluminijevi zlitini.

compared to steel RAVNEX HD due to a thinner reaction layer and a smaller fraction of cracks.

For comparison, the wear resistance of UTOPMO1 in AlSi12 aluminium alloy at a temperature of 600 and 670 °C was tested (Fig. 5). Reaction layer of sample 5 (lower temperature) is formed from thin end even layer. A much thicker reaction layer is seen by sample 6 (higher temperature), which is composed at least from three different layers. It was estimated that the proportion of iron in aluminium near the phase boundary is increased due to better diffusion of iron, whereas at least one layer contains also higher portion of silicon.

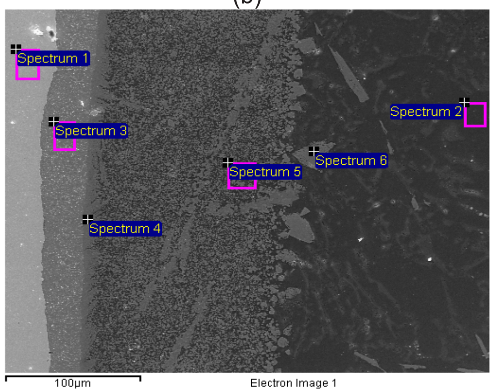
In the case of sample 1 in Fig. 6, the growth of intermetallic Fe-phases in molten aluminium is observed in the form of a tongue. Two different layers, one is compact, slightly brighter and thicker (closer to steel), and the other in the form

(a)



2 (mas.%): Cr 5,7, Mo 2.2, Si 1,1, Al 0,9, Fe ostalo
3 (mas.%): Fe 1,2, Si 15,4, Al ostalo
4 (mas.%):, Fe 26, Si 17,4, Al ostalo
5 (mas.%): Fe 28, Si 16,3, Al ostalo

(b)

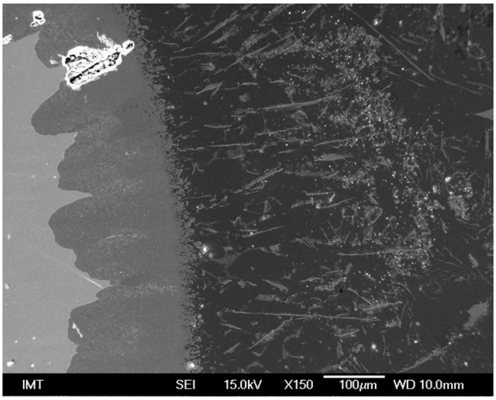


1 (mas.%): Cr 5,3, Mo 1,4, Si 1,1, Al 1,1, Fe ostalo
2 (mas.%): Si 2,7, Al ostalo
3 (mas.%): Fe 42, Cr 2,4, Si 4,5, Al ostalo
4 (mas.%): Fe 27, Cr 2.0, Si 14, Al ostalo
5 (mas.%): Fe 9,9, Cr 1,7, Si 15, Al ostalo
6 (mas.%): Fe 24, Cr 1,4, Si 10, Al ostalo

Sl. 7. Mikroposnetki in EDS analiza interakcijskega sloja v vzorcu 5 (a) in 6 (b)

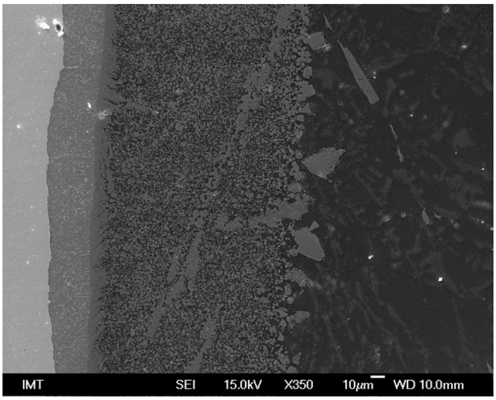
Fig. 7. Micrograph and microanalysis of the interaction layer in the sample 5 (a) and 6 (b)

(a)



UTOP MO1, Al99.7, 700 ºC, 70 min^{-1}

(b)



UTOP MO1, AlSi12, 700 ºC, 70 min^{-1}

Sl. 8. Mikroposnetki interakcijskega sloja v vzorcu 3 (a) in 6 (b)

Fig. 8. Micrograph of the interaction layer in the sample 3 (a) and 6 (b)

Slika 8 predstavlja primerjavo mikrostrukture reakcijske plasti med vzorci 3 in 6; vzorec 3 (UTOP MO1) je bil preizkušan v zlitini Al99,7 in vzorec 6 (UTOP MO1) v zlitini AlSi12. Po debelini, morfologiji in kemični sestavi je reakcijska plast popolnoma drugačna. Formirane Fe-faze v vzorcu 3 so v obliki igel (tipa Al_3Fe) in v vzorcu 6 v obliki globulita (tipa AlFeSi).

of needles or tongue can be distinguished. Analyses represent: 1 - steel composition, 2 - Al-Fe-phase in contact with tool steel with a higher proportion chromium, 3 and 4 - Al-Fe-phase with smaller amount of iron closer to aluminium, 5 - composition of the aluminium and 6 – iron phase in aluminium. The concentration of aluminium is greater in the region of the layer closer to the aluminium alloy, which could be $Al_{13}Fe_4$ phase and decreases when closer to the steel, which could be Al_5Fe_2 phase. The layer closer to aluminium is fragmented, whereas iron dissolves in molten aluminium in the form of needles from Fe-phase.

Fig. 7 represents the microstructure and microanalysis of the sample 5 and 6 where the tool steel UTOP MO1 is in contact with AlSi12 alloy. At sample 5 (Fig. 7a), the reaction layer is thin with a wide layer of dissolved Fe - phases in the alloy AlSi12. At a higher temperature, the wear of the sample is much larger, the reaction layer is composed of three layers as seen from the microstructure and EDS microanalysis. Larger Fe-phases float in aluminium alloy.

Fig 8. represents a comparison of the microstructure of a reaction layer between samples 3 and 6, whereas sample 3 (UTOP MO1) was tested in aluminium 99.7 and sample 6 (UTOP MO1) was tested in AlSi12 alloy at the same temperature and rpm. The reaction zone is completely different regarding to thickness, morphology and chemical composition. Formed Fe-phases in sample 3 are in form of needles (Al_3Fe type) and in sample 6 in a globulite form (AlFeSi type).

4 Conclusions

It can be concluded that the stability of the hot-working tool steel RAVNEX HD and UTOP MO1, which are in contact with molten

4 Zaključek

Sklepamo lahko, da je stabilnost orodnega jekla za delo v vročem RAVNEX HD in UTOP MO1, ki sta v stiku s staljenim aluminijem, odvisna od več parametrov. Med orodnim jeklom in tekočim aluminijem hitro poteka reakcija, medtem ko nastane intermetalna reakcijska plast. Metalografske preiskave so pokazale nastanek intermetalnih Fe-faz, verjetno Al_5Fe_2 in $Al_{13}Fe_4$. Reakcijska cona je večplasten. Najverjetneje se faza Al_5Fe_2 pojavila na fazni meji z orodnim jeklom in je debelejša, medtem ko se faza $Al_{13}Fe_4$ tvori na fazni meji z aluminijem. V reakcijski plasti se raztopijo tudi drugi legirni elementi, kot sta krom in molibden. Poroznost in razpoke opazimo v reakcijski plasti, kar je še posebej opazno pri jeklu RAVNEX HD. Razpoke so bile prisotne tudi v območju aluminija zaradi večje koncentracije železa. Rezultati so pokazali povečano obrabo vzorcev, pri katerih je bilo število vrtljajev višje in/ali je bila temperatura višja. Reakcijska plast ni popolnoma enakomerna; na določenih mestih se lušči in je pri višji temperaturi debelejša. Železo iz orodnega jekla se skozi reakcijsko plast difundira v tekoči aluminij. Pri nasičenju nastajajo železne faze. Opaženo je bilo tudi, da med obrabo orodnega jekla sloj odstopa, posledično so manjši in večji delci železove intermetalne faze in vključkov orodnega jekla prešli v aluminijevo talino.

Zahvala

To delo so podprli Republika Slovenija, Ministrstvo za izobraževanje, znanost in šport ter Evropska komisija za evropski regionalni razvoj in je bilo narejeno v okviru programa Materiali in tehnologije za nove aplikacije (MARTINA, številka nepovratnih sredstev: C3330-16-529008).

aluminium, depends on several parameters. A reaction takes course rapidly between the tool steel and the liquid aluminium, whereas the intermetallic reaction layer forms. Metallographic investigations have demonstrated the formation of intermetallic Fe-phases, presumably Al_5Fe_2 and $Al_{13}Fe_4$. The reaction cone is multilayer. Presumably, phase Al_5Fe_2 appeared on the phase boundary with tool steel and is thicker, whereas phase $Al_{13}Fe_4$ forms on the phase boundary with aluminium. In reaction layer also other alloying elements like chromium and molybdenum dissolved. Porosity and cracks are observed in the reaction layer, which is especially noticeable for RAVNEX HD steel. Cracks were also present in the aluminium region due to the concentration gradient of iron. The results showed increased wear on samples where the rpm was higher and/or the temperature was higher. The reaction layer is not completely uniform; in certain places it is departed and is thicker at a higher temperature. Iron from tool steel diffuses through the reaction layer into liquid aluminium. At the saturation, iron phases are formed. It was also observed that during the wear of the tool steel, the layer departs. Consequently, the smaller and larger particles of the iron intermetallic phase and the inclusion of the tool steel go into the aluminium melt.

Acknowledgements

This work was supported by the Republic of Slovenia, the Ministry of Education, Science and Sport, by the European Commissionand the European Regional Development. This work was made within a frame of the program Materials and Technologies for New Applications (MARTINA, grant number: C3330-16-529008).

Reference

1 R. A. Mesquita, Tool Steels: Properties and Performance, CRC Press, Taylor & Francis Group, 2017, pp.258.

2 Steelselector: Steel SINOXX 4542, SIJ Metal Ravne, d.o.o., 2016, Accessible on Internet: https://steelselector.sij.si/steels/PK346.html,

3 A. Persson, J. Bergstrom, Influence of surface engineering of the performance of tool steels for die casting, Proceedings of the 6th International Tooling Conference: the use of tool steels: experience and research, (J. Bergström, G. Fredriksson, M. Johansson, O. Kotik, F. Thuvander), Karlstad University, 10-13 September 2002., Karlstad, Sweden, pp. 1003.

4 K. N. Obiekea, S. Y. Aku, D. S. Yawas, Effect of pressure on mechanical properties and microstructure of die cast aluminium A380 alloy, Journal of minerals and materials characterization and engineering, 2(2014), pp.248.

5 N. Tang, Experimental and theoretical research in interfacial reaction of solid Co with molten aluminium and its alloys, Transactions of nonferrous metals society of China, 25(2015)6, pp. 1715.

6 M. Yan, Z. Fan, Durability of materials in molten aluminium alloys, Journal of materials science, 36(2001), pp. 285.

7 F. Yin, M. Zhao, Y. Liu, Z., Li, Effect of Si on growth kinetics of intermetallic compounds during reaction between solid iron and molten aluminium, Transactions of nonferrous metals society of China, 23(2013)2, pp. 556.

8 S. Hwang, J. Song, Y. Kim, Effects of carbon content of carbon steel on its dissolution into a molten aluminium alloy, Materials science and engineering A, 390(2005)1-2, pp.437.

9 Y. Erarslan, Wear performance of in-situ aluminium matrix composite after micro-arc oxidation, Transactions of nonferrous metals of China, 23(2013)2, pp. 347.

10 J. H. Selverian, A. R., Marder, M. R., Notis, Reaction between solid iron and liquid Al-Zn Baths, Metallurgical Transactions A, 19(1988), pp. 1193.

11 I. I. Danzo, Y. Houbaert, K. Verbeken, Diffusion driven columnar grain growth induced in an Al–Si-coated steel substrate. Surface and coatings technology, 2014, Vol. 251, 15-20

12 A. Molinari, M. Pellizzari, G. Straffelini, M. Pirovano, Corrosion behaviour of a surface-treated AISI H11 hot work tool steel in molten aluminium alloy. Surface and coatings technology, 2000, Vol. 126, no.1, 31-38

13 M. Sundqvist, S. Hogmark, Effects of liquid aluminium on hot-work tool steel. Tribology international, 1993, Vol. 26, no. 2, 129-134

14 A. Bouayad, C. Gerometta, A. Belkebir, A. Ambari, Kinetic interactions between solid iron and molten aluminium. Materials science and engineering: A, 2003, Vol. 363, no. 1-2, 53-61