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25 LET VAKUUMSKE TOPLOTNE IN KEMOTERMIČNE OBDELAVE NA INŠTITUTU ZA KOVINSKE MATERIALE IN TEHNOLOGIJE, LJUBLJANA

25 YEARS OF VACUUM HEAT TREATMENT AND SURFACE ENGINEERING AT THE INSTITUTE OF METALS AND TECHNOLOGY, LJUBLJANA

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Opisan je pregled petindvajset let delovanja na področju vakuumske toplotne in kemotermične obdelave orodnih in hitroreznih jekel. Raziskovalni dosežki ob podpori drugih sodobno opremljenih laboratorijev Inštitutu za kovinske materiale in tehnologije z inovativnim načinom omogočajo spremljanje in uvajanje najsodobnejših postopkov vakuumske toplotne obdelave in inženirstva površin ter njihovo karakterizacijo, ki je relevantna za slovensko industrijo. Pri tovrstnih tehnologijah je pomembno znanje, škodljiv vpliv na okolje je ničen, poraba energije in dragih materialov je majhna, zato je raziskovalno- -razvojna dejavnost na področju vakuumske toplotne in kemotermične obdelave orodnih in hitroreznih jekel v povezavi z orodjarsko, kovinsko predelovalno, jeklarsko in avtomobilsko industrijo zelo primerna za Slovenijo, ki ima na tem področju tradicijo in usposobljene kadre.

Ključne besede: vakuumska toplotna obdelava, inženirstvo površin, orodna jekla, hitrorezna jekla, lomna žilavost

This review describes twenty-five years of operation in the field of vacuum heat treatment and surface engineering of tools and high-speed steels. Research achievements, with the support of other well-equipped laboratories, enables the Institute of Metals and Technology, an innovative approach to monitoring and for the introduction of procedures in the field of vacuum heat treatment and surface engineering and their characterization, which is relevant to Slovenian industry. When such technology is important knowledge, the adverse environmental impact is nil, the energy consumption and the use of expensive material is small, from these points of view the R & D activities in the field of vacuum heat and surface engineering of tools and high-speed steels in conjunction with the tool industry, metal processing and automotive industries is very suitable for Slovenia, which has a tradition in this area as well as the trained staff.

Key words: vacuum heat treatment, surface engineering, tool steels, high-speed steels, fracture toughness

1 RAZVOJ TEHNOLOGIJ VAKUUMSKE TOPLOTNE OBDELAVE IN OBDELAVE POVRšIN S PLAZMO

Na Inštitutu za kovinske materiale in tehnologije je bil leta 1985 na pobudo prof. dr. Franca Vodopivca ustanovljen prvi center za vakuumsko toplotno in kemotermično obdelavo (CVT&KTO) orodnih in hitroreznih jekel v Sloveniji. Investicijo v takrat najsodobnejšo vakuumsko peč IPSEN VTTC-324R (**slika 1**), ki je bila podlaga za tehnološke raziskave na področju brezogljične okolju prijazne in energijsko varčne vakuumske toplotne obdelave orodij, sta omogočila država in Metal Ravne.

Karakteristike peči zagotavljajo optimalne možnosti vakuumske toplotne obdelave najzahtevnejših orodij za delo v vročem, za delo v hladnem in orodij za brizganje plastike.

Takrat in še danes je ta najsodobnejša peč omogočila, da smo s ciljno usmerjenimi R&D projekti slovenskim orodjarnam v najtežjem obdobju njihovega prestrukturiranja omogočili relativno hiter prehod iz konvencio-

1 DEVELOPMENT OF VACUUM HEAT TREATMENT AND PLASMA SURFACE TREATMENT TECHNOLOGY

In 1985 at the Institute of Metals and Technology, at the suggestion of prof. dr. Franc Vodopivec, the first centre for the vacuum heat treatment and surface engineering (VHT & SEC) of tool steels and high-speed steels was established in Slovenia. The investment in the then most-advanced vacuum furnace, the IPSEN VTTC-324R, in **Figure 1**, which was the basis for the technological research in less-carbon, environmentally friendly and energy-saving vacuum heat treatment of tools and dies, was possible thanks to the government and the company Metal Ravne.

The characteristics of the furnace ensure an optimal vacuum heat treatment for the most demanding tools and dies for hot-work and cold-work applications, as well as for plastic injection moulding.

It has remained one of the most modern furnaces and has offered the possibility of targeted R & D projects to Slovenian toolmakers during the toughest period of their

Slika 1: Enokomorna vakuumska peč Ipsen VTTC324-R s homogenim plinskim ohlajanjem pod visokim tlakom
Figure 1: Single-chamber Ipsen VTTC324-R vacuum furnace with homogeneous high-pressure gas quenching

nalne na vakuumsko toplotno obdelavo orodij za kupce, ki so naročila pogojevali z zelo visokimi kakovostnimi standardi, ki so jim lahko zadostili le z najsodobnejšo tehnologijo izdelave orodij.

Pomemben dejavnik, ki vpliva na trajnost orodij, je toplotna obdelava, po kateri orodno jeklo dobi kritične lastnosti, ki povečajo njegovo odpornost proti prevladujočemu mehanizmu nastanka poškodb. Optimalni postopek toplotne obdelave je mogoč v sodobnih vakuumskih pečeh s hitrim ohlajanjem v toku dušika, helija, vodika ali mešanice različnih plinov pod visokim tlakom. Prav uvedba vakuumske toplotne obdelave je omogočila, da so slovenske orodjarne ohranile svoj tržni delež in ga celo povečale med prilagajanjem na bolj zahtevne nove trge, saj so zaradi relativno hitrega prehoda na boljšo tehnologijo toplotne obdelave, ki jo je imel inštitut, dosegle in celo presegle zahtevano vzdržljivost orodij.

Na osnovi izkušenj in dobrih rezultatov s področja vakuumske toplotne obdelave orodnih jekel smo leta 1993 na IMT-ju investirali v prvo plazemsko tehnologijo nitriranja v pulzirajoči plazmi, ki omogoča kontrolirano modificiranje površine kovinskih materialov. Investicijo je IMT-ju omogočilo ministrstvo s finančno pomočjo podjetij KOLEKTOR, d. d., iz Idrije in ORODJARNE GORENJE, d. o. o., iz Velenja. Reaktor za nitriranje v pulzirajoči plazmi METAPLAS-IONON (**slika 2**) je bil izdelan po naših zahtevah in najnovejših spoznanjih o nitriranju kompleksnih orodij in strojnih delov, izdelanih iz jekla, titana in titanovih zlitin.

S kontroliranim modificiranjem delovnih površin orodij in strojnih delov povečamo odpornost proti obrabi, zmanjšamo koeficient trenja, povečamo trajno dinamično trdnost in korozijsko odpornost, s postopkom oksinitriranja pa tudi estetski videz.

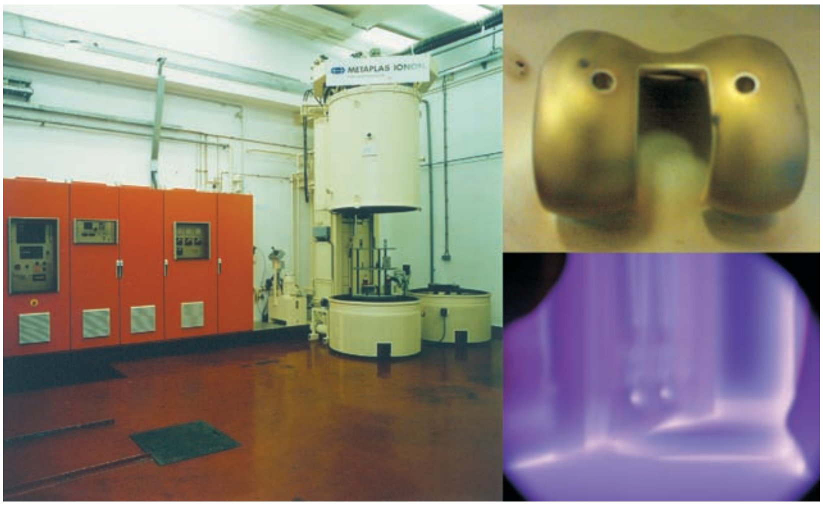
Poleg vrhunskih storitev široke palete kemotermičnih obdelav za slovenske orodjarne in kovinsko predelovalno industrijo je reaktor primeren tudi za raziskovalno in

restructuring and allowed a relatively rapid transition from conventional heat treatment in the vacuum heat treatment of tools and dies for customers who required very high quality standards that can only be met with the latest tooling technology.

One of the most influential factors affecting the sustainability of tools is the heat treatment of the tool steel that ensures the critical properties, thereby increasing the tool's resistance to the dominant damage mechanism for specific working operation when the tool is subjected to stresses. The optimal heat-treatment process is possible only in modern vacuum furnaces with rapid cooling in a stream of nitrogen, helium or hydrogen or in a mixture of different gasses under high pressure. The introduction of the vacuum heat-treatment process has enabled Slovenian toolmakers to maintain, and actually even increase, their market share in the critical period of adaptation to new, more demanding markets. The produced tools reached, and even surpassed, the required lifetime due to the relatively rapid transition to a better heat-treatment technology introduced by the IMT.

On the basis of experience and good results in the field of the vacuum heat treatment of tool and high-speed steels, in 1993 IMT was the first in the country to invest in this pulsed-plasma nitriding technology. This process allows the controlled surface modification of metallic materials. The investment was supported by the Government and the companies KOLEKTOR, d. d., Idrija and ORODJARNA GORENJE, d. o. o., Velenje. The reactor for the pulsed-plasma nitriding METAPLAS-IONON (**Figure 2**) was built according to our requirements and the latest knowledge on the nitriding of complex tools and dies and for steel, titanium and titanium alloy parts.

The controlled nitriding of the working surfaces of tools, dies and machine parts increases the wear resistance, decreases the friction, increases the fatigue strength



Slika 2: Peč za nitriranje v pulzirajoči plazmi METAPLAS-IONON in nitrirana površina dela kolenske proteze iz Ti (desno zgoraj) in nitriranje orodja iz jekla za delo v vročem (desno spodaj).
Figure 2: Furnace for pulsed-plasma nitriding METAPLAS-IONON, the nitrided surface of a Ti knee prostheses and the nitriding of tool steel for a hot-work application.

razvojno dejavnost. To je še posebej pomembno danes, ko so vse večje zahteve po trajnosti orodij za preoblikovanje naprednih visokotrdnostnih jekel (AHSS), za zmanjševanje mase strojnih delov in izboljšanje njihovih lastnosti. Poleg tega omogoča tudi raziskave na področju inženirstva površin, kjer je nitriranje v pulzirajoči plazmi osnova za t. i. dupleksni postopek (PVD ali PACVD), pri katerem nanašamo trdo eno ali večplastno prevleko na nitridno plast, ki ima dovolj veliko nosilnost.

CVT&KTO je sodeloval z več kot 300 orodjarnami in podjetji iz kovinskopredelovalne industrije iz Slovenije in tujine. Danes delujejo v Sloveniji trije komercialni centri za vakuumsko toplotno obdelavo, v nekaterih tovarnah v Sloveniji pa so investirali v tovrstne tehnologije za lastne potrebe. To je trden dokaz, da je bil uspešen prenos teh zahtevnih HT-tehnologij v slovenski prostor. Zaradi dobrih izkušenj tudi v drugih podjetjih za lastne potrebe in še posebej zaradi okoljskih zahtev načrtujejo nove investicije v tovrstne tehnologije.

2 RAZISKOVALNO DELO

Z uvajanjem novih materialov, tudi nanomaterialov, so povezani novi procesi na podlagi plazemskih tehnologij (npr. PACVD, PVD itd.), ki bodo v prihodnjih letih ključne poleg že uveljavljenih vakuumskih tehnologij za kovinskopredelovalna podjetja in orodjarne, ki sodelujejo z avtomobilsko industrijo in bodo hotele ohraniti konkurenčnost. Za prenos tovrstnih tehnologij je za vsako od novo razvitih vrst materialov potreben nov nabor procesnih stopenj in zmogljivosti, nov način karakterizacije mikrostrukture, ki omogoča razumevanje različnih lastnosti in njihove medsebojne vplive. Ta dinamičen, interaktiven razvoj se nadaljuje in zahteva poglobljeno razumevanje vseh dejavnikov, ki so vključeni v uvajanje novih vrst materialov in tehnologij v uporabo. Zato je bil vsa ta leta CVT&KTO tudi raziskovalno ploden, rezultate pa se je trudil čim hitreje presaditi v industrijsko prakso.

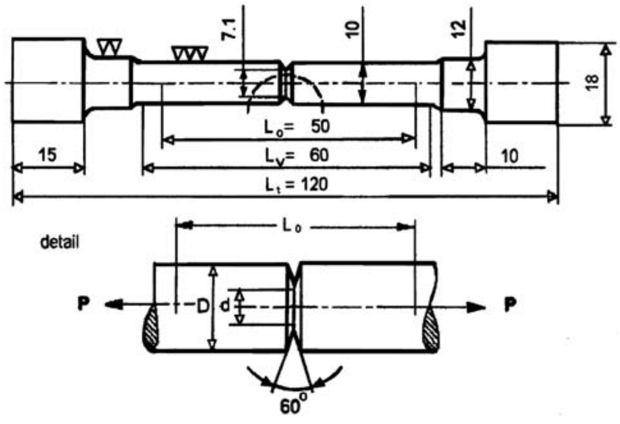
and corrosion resistance and, ultimately, with oxinitriding, the aesthetic appearance can also be improved.

In addition to a wide range of top-quality services of thermo-chemical treatments for Slovenian toolmakers and the metal-processing industry, the reactor is also used for R&D. This is especially important with the increasing focus on the sustainability of tools and dies for the formability of advanced high-strength steels (AHSS), reducing the weight of machine parts and improving their properties. In addition, research activity is possible in the field of surface engineering with the pulsed-plasma nitriding as a basis for duplex processes (PVD or PACVD), which make it possible to apply single or multilayer hard coatings on a nitrided layer to increase the bearing capacity.

By the end of 2009, VHT & SEC had serviced more than 300 tool and metal-processing companies in Slovenia and abroad. During this time in Slovenia three commercial heat-treatment centres have been in operation and some companies have invested in this technology for their own needs. This expanded use confirms that the transfer of vacuum and plasma HT technologies in Slovenia was successful, and accordingly in other enterprises for their own needs and environmental requirements, in particular, new investments in these technologies are planned.

2 RESEARCH WORK

With the introduction of new materials, nano-related materials and new processes based on plasma technologies (such as PACVD, PVD, etc.) will, in the coming years, also become a key addition to the traditional vacuum technologies for metal-processing companies and tool shops, working for the automotive industry, and wishing to remain competitive. For the transfer of such technologies, for each of the newly developed types of materials, a new set of procedural stages and facilities is required and a new approach to the characterization of



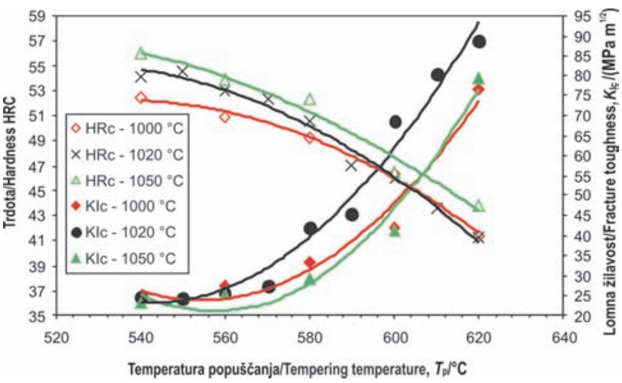
Slika 3: Cilindrični natezni preizkušanec za merjenje lomne žilavosti z zarezo po obodu in utrujenostno razpoko v dnu zareze. Vse dimenzije so v milimetrih.
Figure 3: Circumferentially notched and fatigue-precracked K_{Ic}-test specimen. All dimensions are in milimeters.

Odmeven dosežek doma in v tujini je bil razvoj metodologije merjenja lomne žilavosti K_{Ic} krhkih orodnih in hitroreznih jekel. Pri materialih z nizko duktilnostjo je določevanje lomne žilavosti zelo zahtevno: predvsem je zahtevna in draga izdelava atomsko ostre utrujenostne razpoke v korenu zareze kaljenega in popuščenega standardnega CT-preizkušanca. Problemi, s katerimi se srečujemo pri izdelavi utrujenostne razpoke, so odločujoče pripomogli k iskanju alternativnih preizkusnih metod za določevanje lomne žilavosti. Ena od alternativnih metod določevanja lomne žilavosti, ki smo jo razvili na IMT, je določevanje lomne žilavosti s cilindričnimi nateznimi preizkušanci z zarezo po obodu in utrujenostno razpoko (K_{Ic}-preizkušanec) (**slika 3**), ki jo izdelamo v vrtilno-upogibnem režimu že pred končno toplotno obdelavo [1]. Ena od prednosti K_{Ic}-preizkušancev v primerjavi s CT-preizkušanci je, da dosežemo ravninsko-deformacijsko stanje s preizkušanci manjših dimenzij [1].

microstructures for understanding the various properties and their interrelation. This dynamic, interactive development continues and requires an in-depth understanding of all the factors involved in introducing the use of new types of materials and technologies. For this reason during all those years, VHT & SEC was also active in research and a great effort was dedicated to transplant research results quickly into industrial practice.

A notable achievement at home and abroad was the development of a measurement methodology for the fracture toughness K_{Ic} of brittle tool and high-speed steels. For materials with a low ductility the measurement of fracture toughness is difficult and particularly complex, and especially costly is obtaining the atomically sharp crack in the root of the notches of standard hardened and tempered CT specimens. The main problem encountered in the manufacture of fatigue cracks was the driving force in the search for alternative test methods for the measurement of the fracture toughness. One of such methods, developed at the IMT, was the determination of fracture toughness with circumferentially notched and fatigue-precracked tensile-test specimens (K_{Ic}-test specimens) with the dimensions in **Figure 3**. With the K_{Ic}-test specimens the fatigue crack can be created by rotating-bending loading before the final heat treatment. One of the advantages of such test specimens is that plain-strain conditions can be achieved using specimens with smaller dimensions than those of conventional CT test specimens [1].

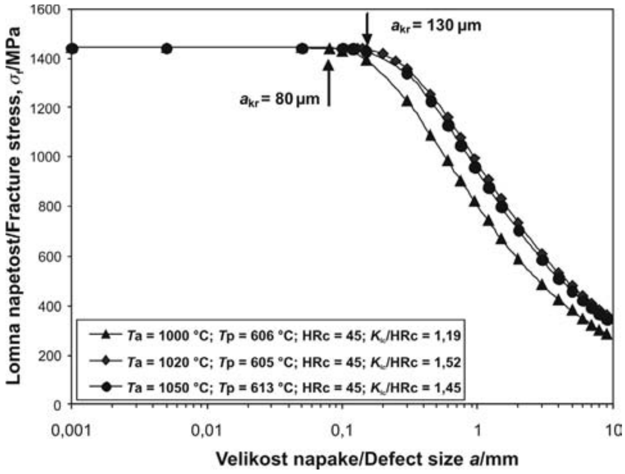
The advantage of the K_{Ic}-test specimens over the standardized CT-specimens (ASTM E399-90) is the radial symmetry, which makes them particularly suited for studying the influence of the microstructure of metallic materials on fracture toughness. The advantage of these specimens is related to the heat transfer, which provides for a completely uniform microstructure. As already mentioned, with the K_{Ic}-test specimens the



Slika 4: Vpliv temperature avstenitizacije in popuščanja na trdoto HRc in lomno žilavost K_{Ic} vakuumsko toplotno obdelanega jekla za delo v vročem H11
Figure 4: Effect of austenitizing and tempering temperatures on the HRc hardness and fracture toughness K_{Ic} of vacuum heat-treated hot-work steel H11

- K_{Ic}-preizkušanec: cilindrični natezni preizkušanec z zarezo po obodu in utrujenostno razpoko v dnu zareze ϕ 10mm x 120 mm
- temperatura avstenitizacije: 1000 °C, 1020 °C in 1050 °C
- čas zadrževanja na temperaturi avstenitizacije: 20 min
- ohlajanje: v toku N_2 pri tlaku 1,05 bar do temperature 100 °C
- parametri ohlajanja $\lambda_{800-500}$: 1,04; 1,02; 1,11
- prvo popuščanje: 2 h pri 540 °C
- drugo popuščanje: 2 h med 540 °C in 620 °C

- K_{Ic}-test specimens: circumferentially notched and fatigue-precracked tensile specimens ϕ 10 mm × 120 mm
- austenitization temperature: 1000 °C, 1020 °C and 1050 °C
- soaking time: 20 min
- quenching: gas quenching in N2 at a pressure of 1.05 bar to 100 °C
- cooling parameters $\lambda_{800-500}$: 1.04; 1.02; 1.11
- first tempering: 2 h at 540 °C
- second tempering: 2 h between 540 °C and 620 °C



Slika 5: Vpliv temperature avstenitizacije na lomne napetosti σ_f za jeklo H11
Figure 5: Influence of austenitizing temperature on the fracture stress σ_f of H11

Prednost takšnih preizkušancev v primerjavi s standardnimi CT-preizkušanci (ASTM E399-90) je tudi njihova radialna simetrija. Zato so še posebej primerni za opredelitev vpliva mikrostrukture kovinskih materialov na lomno žilavost. Izoblikovanje mikrostrukture po obodu je popolnoma enakomerno zaradi radialno simetričnega odvajanja toplote. Pri merjenju lomne žilavosti trdih in krhkih kovinskih materialov, kjer nam zaradi velike zarezne občutljivosti le s težavo, če sploh, uspe izdelati razpoko z utrujanjem, lahko utrujenostno razpoko na tovrstnemu preizkušancu izdelamo še pred končno toplotno obdelavo.

To nam je omogočilo, da smo pri vakuumski toplotni obdelavi orodnih in hitroreznih jekel lomno žilavost K_{Ic} uvedli kot drugi parameter. Tako lahko iz diagrama popuščanja za jeklo, izbrano za specifično operacijo, izberemo najprimernejše razmerje med žilavostjo in trdoto. Taki diagrami popuščanja nam omogočajo, da izberemo parametre vakuumske toplotne obdelave, s katerimi to tudi dosežemo pri izbranem jeklu (**slika 4**).

Na osnovi poznane lomne žilavosti lahko izračunamo kritično velikost napake, ki jo orodno jeklo pri lomni napetosti še prenese (**slika 5**).

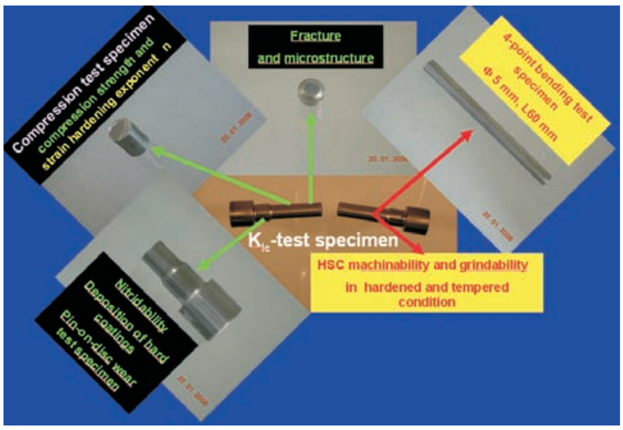
Tudi pri raziskovalnem delu so stroški eksperimentov pomembni, zato po meritvi lomne žilavosti dele K_{Ic}-preizkušancev uporabljamo še za meritve trdote, upogibne

fatigue crack can be created with rotating-bending loading before the final heat treatment.

This allowed us to introduce the fracture toughness K_{Ic} as a second parameter in the vacuum heat treatment of tool and high-speed steels. In this way, from the tempering diagram for the selected steel it is possible to choose the most appropriate relationship between the toughness and the hardness for a specific application. Such a tempering diagram allows us to also choose the parameters of vacuum heat treatment that give the selected steel the desired performance (**Figure 4**).

Knowing the fracture toughness K_{Ic} and the ultimate tensile stress σ_u of the investigated tool steel, the fracture stress σ_f and the critical defects' size in the tool steel can be calculated (**Figure 5**).

The costs of experiments for research projects are important. For this reason, after a determination of the fracture toughness, two parts of the K_{Ic}-test specimens are used to manufacture different samples for the hardness measurements, the four-point bending, the compressive strength and strain hardening exponent determination, the analysis of fracture surfaces and of the microstructure and for the specimens of other tribological and technological tests, necessary for understanding the behaviour of tool steels and high-speed steels in practice (**Figure 6**).



Slika 6: Možnosti izdelave različnih preizkušancev iz K_{Ic}-preizkušanca
Figure 6: Possibilities for the manufacturing of various trial specimens from the K_{Ic}-test specimens

trdnosti, tlačne trdnosti, določitev eksponenta deformacijskega utrjevanja, analizo prelomnih površin, mikrostrukturno analizo ter za izdelavo preizkušancev za druge tribološke in tehnološke preizkuse, ki so pomembni za razumevanje vedenja orodnih in hitroreznih jekel v praksi (**slika 6**).

Ker so vse meritve izvedene na istem preizkušancu, lahko za različne lastnosti ugotovimo, v kakšni medsebojni povezavi so posamezne lastnosti.

Odmeven znanstveni dosežek je razvoj polempirične enačbe (1), ki omogoča, da na osnovi trdote Rockwell-C, volumskega deleža zaostalega avstenita f_{aust}, volumskega deleža neraztopljenih evtektičnih karbidov f_c, srednje razdalje med temi karbidi d_p, kumulativnega deleža $f_{c\geq a_{crit}}$ karbidov in/ali karbidnih skupkov, ki so $\geq a_{crit}$ (a_{crit}, kritična velikost napake) in modula elastičnosti E izračunamo lomno žilavost za hitrorezna in ledeburitna jekla s trdoto HRc večjo od 57 HRc [2].

$$K_{Ic}=1{,}363\cdot\left(\frac{HRc}{HRc-53}\right)\cdot \left[\sqrt{Ed_p}\cdot(f_c)^{-\left(\frac{1}{6}\right)}\cdot(1-f_{c\geq a_{crit}})\cdot(1+f_{aust})\right]\left[MPa\sqrt{m}\right] \quad (1)$$

Na osnovi te enačbe je mogoče teoretično pojasniti vpliv mikrostrukture hitroreznih in ledeburitni jekel na lomno žilavost. Namreč, s toplotno obdelavo jeklu s spreminjanjem mikrostrukture spreminjamo lastnosti.

Za orodni jekli za delo v vročem H11 in H13 je bila razvita empirična enačba (2) s katero na osnovi udarne žilavosti Charpy-V in trdote Rockwell-C ocenimo lomno žilavost orodnega jekla:

$$K_{Ic}=4{,}53\cdot CVN^{1{,}11}\cdot HRc^{-0{,}135}\left[MPa\sqrt{m}\right] \quad (2)$$

kjer je CVN absorbirano udarno delo in HRc trdota Rockwell-C [3].

Na **sliki 7** je prikazano ujemanje med eksperimentalno določeno lomno žilavostjo in lomno žilavostjo, izračunano na osnovi enačbe (2) za jekli H11 in H13.

Tovrstne ocene so še posebej pomembne pri analizi vzrokov nastanka poškodb orodja (npr. prelom orodja med obratovanjem itd.), pri katerem ni mogoča izdelava

Since all the measurements are performed on the same test specimen, it is possible to find the correlation between different characteristics for individual properties.

A scientifically notable achievement was the development of the semi-empirical equation (1) that makes it possible to calculate the fracture toughness of high-speed and ledeburitic steels [2] on the basis of the modulus of elasticity E, the mean distance between the undissolved eutectic carbides, d_p, the Rockwell-C hardness, f_c and f_{aust} as the volume fractions of undissolved eutectic carbides and retained austenite, and $f_{c\geq a_{crit}}$ as the cumulative fraction of undissolved eutectic carbides and/or carbide clusters equal to, or larger than, the critical defect size ($\geq a_{crit}$), with a Rockwell-C hardness higher than 57 HRc.

$$K_{Ic}=1{,}363\cdot\left(\frac{HRc}{HRc-53}\right)\cdot \left[\sqrt{Ed_p}\cdot(f_c)^{-\left(\frac{1}{6}\right)}\cdot(1-f_{c\geq a_{crit}})\cdot(1+f_{aust})\right]\left[MPa\sqrt{m}\right] \quad (1)$$

On the basis of this equation it is theoretically possible to determine the influence of individual constituents of the microstructure of high-speed and ledeburitic steels on the fracture toughness. Namely, the heat treatment can modify the material properties by changing the microstructure.

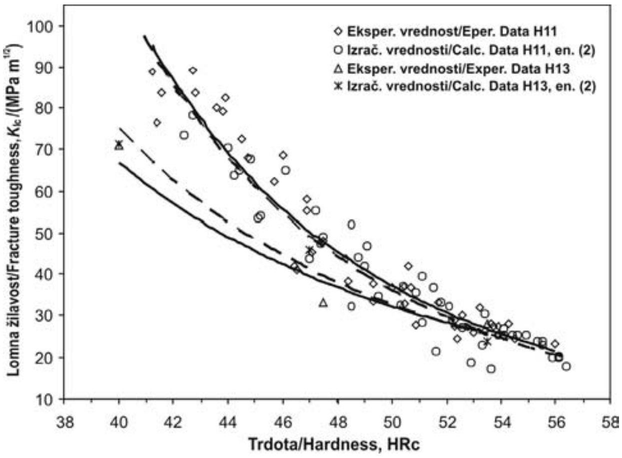
For the hot-work tool steels H11 and H13, an empirical equation based on the Charpy-V impact toughness and the Rockvell-C hardness was developed (2), to estimate the fracture toughness of tool steels, respectively:

$$K_{Ic}=4{,}53\cdot CVN^{1{,}11}\cdot HRc^{-0{,}135}\left[MPa\sqrt{m}\right] \quad (2)$$

where CVN is the energy absorption and HRc is the Rockwell-C hardness [3].

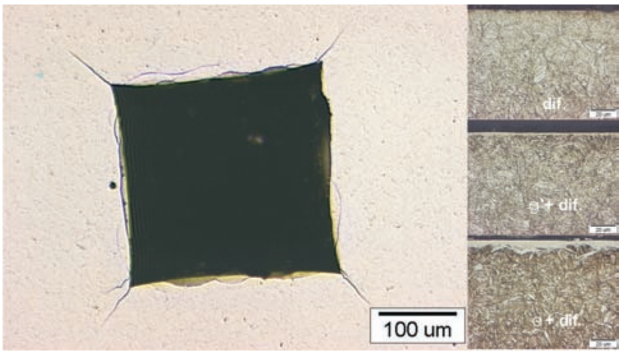
Figure 7 shows the correlation between the fracture toughness of the experimentally determined and calculated fracture toughness on the basis of equation (2) for H11 and H13 steels.

Such assessments are particularly important for the analysis of the failures for tools (e.g., tool cracking



Slika 7: Primerjava med eksperimentalno določenimi vrednostmi lomne žilavosti K_{Ic} in izračunanimi pri enaki trdoti za jekli H11 in H13 iz *CVN* udarne žilavosti in trdote HRc z enačbo (2)

Figure 7: Comparison between the experimentally obtained values of K_{Ic} and the pertained hardness, and the values calculated for the investigated hot-work tool steels H11 and H13 from CVN test results and the pertained hardness (eq. 2)



Slika 8: Vickersov vtis na krhki nitridni plasti; Palmqvist-ove razpoke, difuzijska plast, spojinska plast γ' in/ali ε [5]
Figure 8: Vickers indentation on brittle nitride layer; Palmqvist cracks, diffusion layer, compound layer of γ' and/or ε [5]

standardnih ali nestandardnih preizkušancev za določevanje lomne žilavosti. Možno pa je izrezati metalografski obrus pri hitroreznih in ledeburitnih jeklih ali *CVN*-preizkušanec pri orodnem jeklu za delo v vročem.

Na področju modificiranja delovnih površin orodij z nitriranje je pomemben razvoj metodologije merjenja lomne žilavosti nitridne plasti z metodo vtiskovanja Vickersove piramide pri različnih obtežbah (**slika 8**).

Za relativno tanke krhke plasti na relativno žilavi podlagi, kot so različne nitridne plasti na orodnem jeklu, smo ugotovili, da je za oceno lomne žilavosti primerna enačba, ki jo je razvil Shetty [4] na osnovi pojava Palmqvistovih razpok [5]:

$$K_{\mathrm{Ic}} = 0{,}0319\left(\frac{P}{al^{1/2}}\right)\left[\mathrm{MPa}\sqrt{m}\right] \qquad (3)$$

kjer je P obtežba, a je polovica srednje dolžine diagonale in $l = \frac{1}{4}\sum_{i=0}^{4} l_i$ je srednja dolžina razpok [6].

Pomemben rezultat raziskav na področju nitridnih plasti je ugotovitev, da lahko na osnovi izmerjenega profila mikrotrdote določimo globino maksimalnih tlačnih napetosti, kar omogoča optimizacijo postopka nitriranja, tako da globino maksimalnih napetosti dosežemo v področju maksimalnega Hertzovega tlaka, kar povečuje trajno nihajno trdnost (**slika 9**) [7].

Podeljeni so bili tudi trije patenti, in sicer za "Indukcijsko ogrevano celico za toplotno in kemotermično obdelavo kovin v zvrtinčeni plasti: patent SI 9800119"; "Dvostranski lamelni skobelni nož za obdelavo kovin, during operation, etc.) in the case when it is not possible to manufacture standard CT or non-standard cylindrical specimens for the fracture toughness measurement. But it is possible to cut out the metallographic sample in the case of high-speed or ledeburitic steels and CVN specimens in the case of hot-work tool steel.

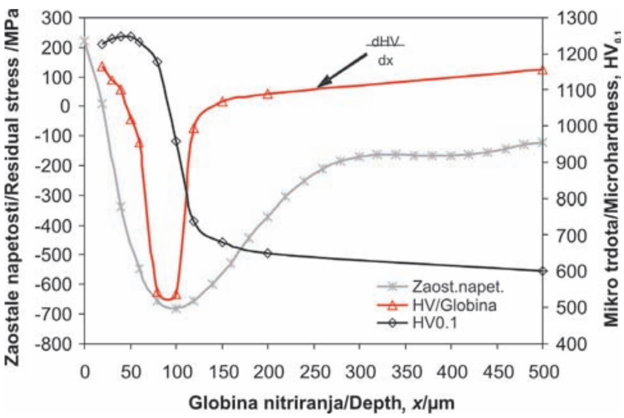
For the modified working surfaces of tools and dies with nitriding, of essential importance is the method of measuring the fracture toughness of the nitride layers with Vickers indentations at various loads (**Figure 8**).

For a relatively thin and brittle layer on a relatively tough substrate, as, for example, the different nitride layers (**Figure 8**) on tool steel, we found that for the assessment of the fracture toughness the Shetty [4] equation (3), developed on the basis of Palmqvist cracks, is appropriate [5]:

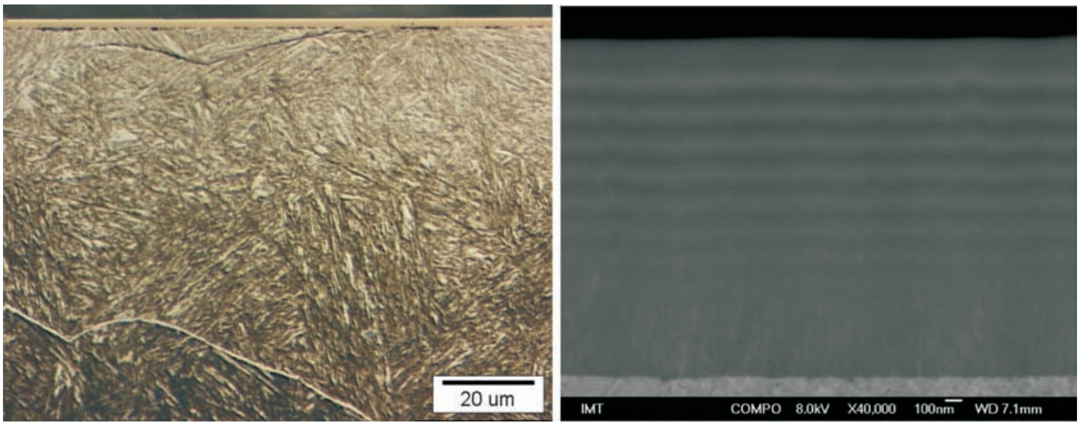
$$K_{\mathrm{Ic}} = 0{,}0319\left(\frac{P}{al^{1/2}}\right)\left[\mathrm{MPa}\sqrt{m}\right] \qquad (3)$$

where P is the load, a medium-length, half of the diagonal and $l = \frac{1}{4}\sum_{i=0}^{4} l_i$ the mean length of the cracks [6].

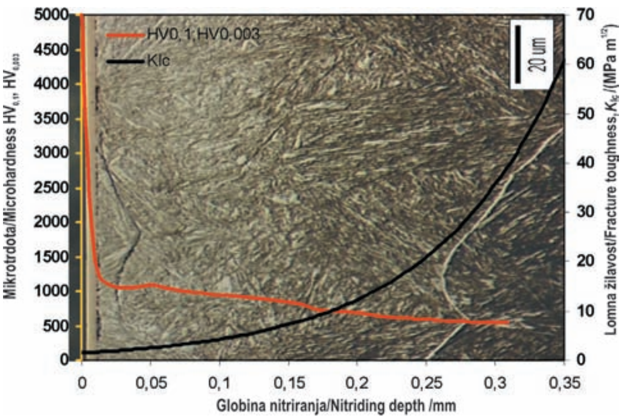
For nitride layers, it is important that the depth of the maximum compressive stresses can be determined on the basis of the measured microhardness profile. This makes it possible to optimise the nitriding process and ensures that the depth of the maximum compressive stresses is achieved in the area of the maximum Hertzian pressure and the obtaining of an increase in the fatigue strength (**Figure 9**) [7].



Slika 9: Primerjava profila mikrotrdote, njegovega odvoda po globini in izmerjenega profila zaostalih napetosti na nitriranem jeklu za delo v vročem H11
Figure 9: Comparison of microhardness depth profile, its derivation over depth and the residual stress profile for the plasma-nitrided hot-work tool steel H11



Slika 10: PACVD-dupleksna večplastna trda prevleka TiB_2/Ti-B-N (21plasti)/TiN na jeklu za delo v vročem H11
Figure 10: Duplex multilayer PACVD hard coating TiB_2/Ti-B-N (21 layers) / TiN on hot-work tool steel, H11



Slika 11: Profil mikrotrdote $HV_{0,1}$ in $HV_{0,003}$ ter lomne žilavosti K_{Ic}, nitriranega vzorca iz jekla H11 z nanosom večplastne trde prevleke iz TiB_2/Ti-B-N/ TiN, nanesene po dupleksnem PACVD- postopku
Figure 11: Micrograph showing HV microhardness depth profile of duplex-treated H11 substrate at a load of 1N and 0.3 N and the corresponding fracture toughness K_{Ic} of the TiB_2/Ti-B-N/ TiN multilayer hard coating.

lesa itd., sestavljen iz nosilne lamele iz konstrukcijskega jekla in dveh enako dolgih, vendar ožjih in tanjših lamel iz kabidne trdine WC-Co: patent SI 9600034 A" in "Povečanje trdote površine zlitine FeAl v masnih deležih (%) s postopkom nitriranja v pulzirajoči plazmi: patent SI 960001"4.

Najnovejše raziskave so usmerjene na področje podhlajevanja orodnih jekel v tekočem dušiku in nanašanja trdih prevlek po dupleksnem postopku PACVD, ki je ključen za trajnost velikih kompleksnih orodij za oblikovanje naprednih, visokotrdnostnih jekel (AHSS), katerih trdnost presega 800 MPa (**slika 10**) [8].

Pri preoblikovanju AHSS-jekel je pomembno, da pri orodju optimiramo sistem: trda prevleka /modificirana površina/ orodno jeklo z ozirom na kritične lastnosti, tj. trdota, razteznost, žilavost in omogočimo doseganje lastnosti, ki povečujejo odpornost sistema proti prevladujočemu mehanizmu nastanka poškodb (obraba, plastična deformacija, krušenje, prelom in lepljenje) (**slika 11**).

3 SKLEP

Slovenske orodjarne in podjetja s področja kovinskopredelovalne industrije, ki sodelujejo z avtomobilsko industrijo, imajo relativno široke programe, vendar pa je

The following patents were awarded: for the "Induction heated cell for heat treatment and thermo chemical treatment of metals in fluidized bed: Patent SI 9800119", for the "Lamellar bilateral planning knife for treatment metal, wood, etc. consisting of carrier strip of construction steel and two equally long but narrow and thin strips of WC-Co hard metal: Patent SI 9600034 A" and for the "Increase the surface hardness of FeAl alloy with the pulsed plasma nitriding: Patent SI 9600014".

Recent research has focused on the area of the deep-cryogenic treatment of tool steels and the application of hard coatings deposited by the PACVD duplex process, which is crucial for the sustainability of large and complex tools for the forming of advanced high-strength steels (AHSS), whose strength exceeds 800 MPa (**Figure 10**) [8].

For the forming of AHSS steels, it is important that the system of working surfaces, i.e., hard coating / modified surface / tool steel of the tool are optimised with respect to the critical properties, i.e., hardness / ductility / toughness, that increase the resistance to the dominant damage mechanism (adhesion, galling, abrasion, plastic deformation, chipping and cracking) (**Figure 11**).

vse manj področij, kjer lahko ohranijo ali na novo pridobijo konkurenčne prednosti. Zato se vse bolj pojavlja potreba po prestrukturiranju in povezavah, kajti le dovolj veliki in uspešni subjekti, ki so povezani s centri znanja, lahko obvladujejo trg in zmorejo velika vlaganja v znanje in razvoj proizvodov z večjo dodano vrednostjo ter v posodobitev opreme, da obdržijo konkurenčnost in zagotovijo akumulacijo, s katero pokrijejo stroške in zagotovijo lastnikom zanimiv profit.

Tehnologije in metodologije, ki jih uvajamo in razvijamo ter so podprte z ekspertnim znanjem drugih laboratorijev na IMT, spadajo v HT-tehnologije in so primerljive s tistimi, ki imajo od tri- do petkrat večjo dodano vrednost.

Ta kratka predstavitev dejavnosti in raziskovalnih dosežkov je dokaz, da CVT&KTO ob podpori drugih sodobno opremljenih laboratorijev za karakterizacijo na IMT z inovativnim načinom omogoča spremljanje in uvajanje najsodobnejših postopkov s področja vakuumske toplotne obdelave in inženirstva površin in njihovo karakterizacijo. Če k temu dodamo še dejstvo, da se pri tovrstnih tehnologijah prodaja predvsem znanje, da je ničen škodljiv vpliv na okolje, poraba energije in dragih materialov pa majhna, je logičen sklep, da je R&D-dejavnost v povezavi z orodjarsko, kovinskopredelovalno in avtomobilsko industrijo, zelo primerna za Slovenijo, ki ima na tem področju tradicijo in usposobljene kadre.

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3 CONCLUSION

Slovenian toolmakers and companies in the metal-processing industry working for the automotive industry have relatively large programs, but in fewer areas can they maintain or regain a competitive advantage. Therefore, there is a growing need for restructuring and connections, because only large enough and successful entities associated with centres of knowledge can be successful in the market and are capable of a significant investment in knowledge and the development of products with higher added value and updated equipment to maintain competitiveness and to ensure the accumulation, which cover the costs and provide an attractive profit for owners.

The introduced and developed technologies and methodologies are also supported by the expert knowledge of other laboratories at the IMT, are part of HT technology and comparable to those of three to five times higher added value.

This brief presentation of the activity and achievements shows that VHT & SEC, with the support of other well-equipped IMT laboratories for characterization, and an innovative approach make it possible to monitor and implement cutting-edge procedures of vacuum heat treatment and surface engineering and their characterization. If the fact is added that in these technologies, it is mainly knowledge that is being sold, the environmental impact is practically zero and the consumption of energy and precious material is small, the conclusion is logical that the R & D activity related to the tool-making industry, the metal-processing and automotive industries, is very appropriate and promising for Slovenia, which has trained staff and a tradition in this area.