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Fracture Toughness of High Strength Steels for Parts Prepared by Metal Injection Molding

Lomová houževnatost vysokopevnostních ocelí pro díly vyrobené injektováním do formy

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Metal injection molding (MIM) is a metalworking process, in which finely-powdered metal is mixed with a binder material to create a "feedstock" that is then shaped and solidified using injection molding. The materials produced in this way can have better usable and mechanical properties than materials produced by a standard metallurgical process. Fracture toughness is expressed in terms of parameters such as K_{Ic}, critical J-integral. The fracture toughness of 4 types of steel was determined. The mechanical properties of tested materials are relatively high, especially the ultimate strength. The materials are relatively brittle, and the K_{Ic} value itself may not provide large differences. The J-integral value, which takes into account the plastic behavior of materials may provide more precise results. The experimental testing was focused on instrumented impact toughness and fracture toughness. Knowledge of the fracture properties allows calculations of the residual life of structures containing a subcritical defect.

***Key words**: Fracture toughness; metal injection molding; mechanical properties; high strength steel*

Metal injection molding (MIM) je proces výroby kovů, ve kterém se jemný práškový kov mísí s pojivem, čímž se vytvoří "surovina", která se následně tvaruje a tuhne za použití vstřikování. Proces MIM dovoluje tvarovat v jediném kroku objemné a složité díly. Takto vyrobené materiály mohou mít lepší užitné a mechanické vlastnosti než materiály vyráběné standardními metalurgickými procesy. Uvedené materiály jsou v průmyslu široce používány jako součásti speciálních konstrukcí. Z materiálových vlastností bylo testování zaměřeno na lomovou houževnatost. Lomová houževnatost znamená odolnost materiálu vůči vzniku a následnému růstu trhliny. Vychází z mechaniky lomu a je v podstatě materiálovou charakteristikou, pomocí níž lze vypočítat nosnost konstrukčního prvku. Z praktického hlediska lze houževnatost charakterizovat jako schopnost materiálu absorbovat energii před tím, než porušení dosáhne určitého mezního stavu. Cílem zkoušky lomové houževnatosti je sledovat odezvu materiálu na přítomnost vady ve standardním zkušebním tělese obsahujícím nacyklovanou únavovou trhlinu. Výsledek je vyjádřen pomocí parametrů houževnatosti, jako je K_{Ic}, J-integrál nebo kritické rozevření trhliny. Sledována byla lomová houževnatost čtyř typů oceli vyrobených MIM technologií. Materiály vyrobené MIM technologií mají poměrně vysoké mechanické vlastnosti, zejména maximální pevnost. Vzhledem k vysoké pevnosti jsou materiály relativně křehké a samotná hodnota houževnatosti K_{Ic} nemá velký rozptyl hodnot. Hodnota J-intergrálu, která bere v úvahu plastické chování materiálů, je rovněž předmětem výzkumu. Samotné testování bylo zaměřeno na houževnatost a houževnatost s výpočtem J-integrálu. Znalost lomových vlastností umožňuje, aby konstrukční součást byla správně dimenzována a tím se zlepšila životnost. Lomové vlastnosti také umožňují výpočet zbytkové životnosti struktur obsahujících podkritickou vadu. Vzhledem k nárůstu použití MIM technologie pro výrobu konstrukčních prvků je velmi důležité tyto vlastnosti prověřovat.

***Klíčová slova**: Lomová houževnatost; vstřikolisování kovů do formy; mechanické vlastnosti; vysokopevnostní oceli*

In metallurgy and material science, fracture toughness refers to a property, which describes the ability of a material containing a crack to resist further fracture. Fracture toughness is a quantitative way of expressing a material's resistance to brittle fracture when a crack is present. If a material has high fracture toughness, it is more prone to ductile fracture. Brittle fracture is characteristic of materials with less fracture toughness. Fracture toughness values may serve as a basis for material comparison, selection, structural flaw tolerance assessment or quality assurance. A large amount of knowledge of failures and fractures, collected from both practice and material testing, contributed to the creation of a separate technical field - fracture mechanics. From basics, the fracture mechanics can be divided to: Linear elastic fracture mechanics (LEFM), applicable for the stress of bodies in the elastic strain area and Elastic-plastic fracture mechanics (EPFM), extended to stress in the elastic-plastic strain area [1]. The theory of Linear Elastic Fracture Mechanics (LEFM) has been developed using a stress intensity factor (K) determined by the stress analysis, and expressed as a function of stress and crack size. The elements of fracture mechanics may be summarized in the form of a triangle having the 3 critical parameters situated at each apex: working stress, fracture toughness and critical flaw size. If two of the three parameters are known, the third can be calculated. Elastic-plastic mechanics (EPFM) theory uses the J-Integral concept first proposed by [1] as a path independent integral for characterizing crack tip stresses and strains [1 – 3]. All metallic materials and alloys that are commercially used in structural parts manifest some plastic deformation in the area of the crack tip when they are subjected to applied stress. The amount of plastic deformation at the crack tip is directly related to the material fracture toughness (the material's ability to resist cracking) and for a given material it varies as a function of part thickness [2, 4]. Metal injection molding (MIM) is a metalworking process, in which finely-powdered metal is mixed with a binder material to create a "feedstock" that is then shaped and solidified using injection molding. The molding process allows high-volume complex parts to be shaped in a single step. After molding, the part undergoes conditioning operations to remove the binder (debinding) and densify the powders. Finished products are small components used in many industries and applications [5 – 7]. There is a broad range of materials available when utilizing the MIM process. Traditional metalworking processes often involve a significant amount of material waste, which makes MIM a highly efficient option for the fabrication of complex components consisting of expensive/special alloys [6].

The research was aimed at a comparison of fracture properties of 4 high strength steels prepared by MIM. Comparison of fracture properties was carried out as in the area of linear elastic fracture mechanics – value K_{Ic} (K_q) and area of elastic-plastic fracture mechanics value J-integral. These values and their achievement are discussed in this article.

1. Materials and methods

2.1 Specimens

The materials used in this study present 4 types of commercial steels produced by Metal injection molding (MIM) technology. Types of steels with basic mechanical properties are listed in Tab. 1.

Tab. 1 Designation of used steels and their basic material properties
Tab. 1 Označení použitých ocelí a jejich mechanické vlastnosti

Steel	DIN Designation	Hardness HRC	Ultimate strength	Yield strength
			(MPa)	
16 720	17Ni4CrMo	43 – 47	1,480	1,180
15 142	42CrMo4	40 – 45	1,420	1,280
14 260	54SiCr6	45 – 47	1,560	1,190
1.7765	32CrMoV12-10	45 – 49	1,400	1,190

In table 2 is shown chemical composition mentioned steels in wt%.

Tab. 2 Chemical composition of used steels (wt. %).
Tab. 2 Chemické složení daných ocelí (hm.%)

Steel	C	Si	Mn	Cr	Mo	Ni
16 720	0.14 - 0.21	0.17 - 0.37	0.25 -0.55	1.35 - 1.65	---	---
15 142	0.38- 0.45	0.17 - 0.37	0.50 - 0.80	0.9 - 1.2	0.15 - 0.30	max 0.5
14 260	0.50 - 0.60	1.30 - 1.60	0.50 - 0.80	0.50 -0.7	---	max 0.5
1.7765	0.30 - 0.35	≤ 0.35	≤ 0.60	2.8 - 3.2	0.80 - 1.20	---

Generally valid specifications for configuration and sample preparation are given in section 7 [2]. All specimens were pre-cracked in three-point bending fatigue based upon the force P_m, as follows [1]:

$$P_m = \frac{0.5 B b_0^2 \sigma_Y}{S}, \tag{1}$$

where B is width of specimen, b_0 is original remaining ligament and σ_y is effective yield strength.

Fatigue pre-cracking requirements have not been fully complied with due to experimental measurement.

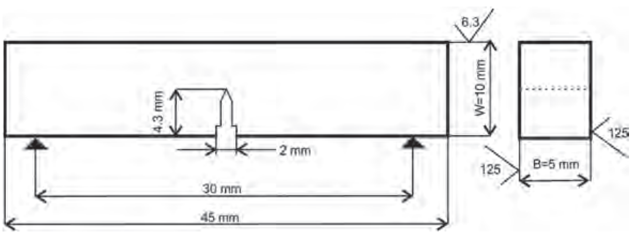

Fig. 1 Drawing of specimen geometry
Obr. 1 Geometrie vzorků

The real dimensions of specimens are showed in Fig. 1 W = 10 mm, B = 5 mm, l = 45 mm, notch length 4.1mm span 30 mm and length of fatigue crack was set to a = 1.3 mm. Exact lenght of fatigue crack was measured after test. Measured characteristic during the test was COD (crack opening displacement in mm). Calculations of K for bend specimen at a force $P_{(i)}$, calculate K as follows:

$$K_{(I)} = \left[\frac{P_i S}{(B B_N)^{1/2} W^{3/2}}\right] f(a_i/W), \tag{2}$$

where:

$$f\left(\frac{a_i}{W}\right) = \frac{3\left(\frac{a_i}{W}\right)^{3/2}\left[1.99-\left(\frac{a_i}{W}\right)\left(1-\frac{a_i}{W}\right)\left(2.15-3.93\left(\frac{a_i}{W}\right)+2.7\left(\frac{a_i}{W}\right)^2\right)\right]}{2\left(1+2\frac{a_i}{W}\right)\left(1-\frac{a_i}{W}\right)^{3/2}}. \tag{3}$$

For the single edge bend specimens, the value of J is calculated as follows:

$$J = J_{el} + J_{pl}, \tag{4}$$

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where J_{el} is elastic component of J and J_{pl} is plastic component of *J*.

J calculations for the basic test method – at a point corresponding to υ and *P* on the specimen force versus displacement record, calculate the J integral as follows:

$$J = \frac{K^2(1-\upsilon^2)}{E} + J_{pl}, \tag{5}$$

where *K* is the value from eq. (2) with $a = a_0$ and

$$J_{pl} = \frac{\eta_{pl} A_{pl}}{B_N b_0}, \tag{6}$$

where A_{pl} is area under force versus displacement record and $\eta_{pl} = 1.9$ if the load-line displacement is used for $A_{pl} = 3.667 - 2.199\ (a_o/W) + 0.437\ (a_o/W)^2$ if the crack mouth opening displacement record is used for A_{pl}, B_N is net specimen thickness ($B_N = B$ if no side grooves are present), and $b_o = W - a_o$.

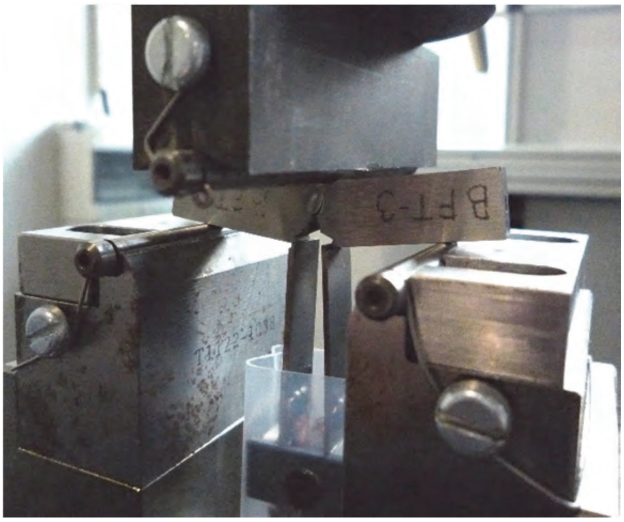

Fig. 2 Photo of the tested specimen in the testing machine
Fig. 2 Fotografie testovaného vzorku ve zkušebním stroji

There is another method for evaluation of *J*-integral based on resistance curve test method [2]. The method has more differences from the above mentioned method.

For general use, it is better to use the basic method of calculations. All fracture toughness tests were carried out on the servohydraulic mechanical testing machine (INSTRON 8802) at room temperature. Fig. 2 shows the broken specimen.

2. Results and discussion

Table 2 shows tested fracture material properties. Table combines fracture toughness value, J-integral value and Charpy impact energy. Other types of showed results are graphical dependence of load vs. COD. There are listed only typical plots for each steel. In Tab. 2 there are listed average values of test results. Fracture toughness test for each material was repeated 5 times. Charpy impact test was repeated 6 times for each material.

Tab. 3 Results of material testing
Tab. 3 Výsledky testování

Steel	K_Q	st. deviation K_Q	J_Q - integral	st. deviation J_Q	Charpy impact energy
	(MPa·m$^{1/2}$)		(kJ·m^{-2})		(J)
16 720	69	3,4	103	11,3	119
15 142	72	4,1	44	8,6	53
14 260	65	18,1	10	4,9	19
1.77656	74	3,05	20	3,02	32

Differences between results for one material were minimal with normal statistical deviation. From fracture toughness point of view, the value K_q with unit MPa·m$^{1/2}$ is written. However, all measurements did not fulfil the condition of plane deformation. This must be specified as K_q. The value $2.5(K_Q/\sigma_{YS})^2$, where σ_{YS} is the 0.2 % offset yield strength in tension [8], shall be calculated. If this quantity is less than the specimen ligament size, *W–a* then K_Q is equal to K_{Ic}. Otherwise, the test is not a valid K_{Ic} test. Expressions for calculating K_Q are given in the standard for each specified specimen configuration. If the test result fails to meet the requirements of standard, it will be necessary to use a larger specimen to determine K_{Ic}. [8, 9]. The minimum K_{ic} value can be specified for production quality control and for acceptance of its standard. If the product is of sufficient size, the K_{Ic} value may be considered as representative. The specification of K_{Ic} values in relation to a particular application should signify that a fracture control study has been conducted for the component in relation to the expected loading and environment, and in relation to the sensitivity and reliability of the crack detection procedures that are to be applied prior to service and subsequently during the anticipated service life [9, 10].

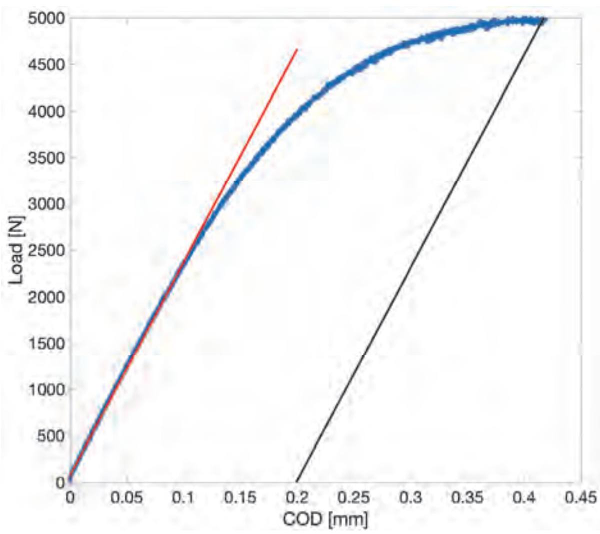

Fig. 3 Dependence of load on COD plot of 1672 steel
Obr. 3 Závislost síly na COD u oceli 1672

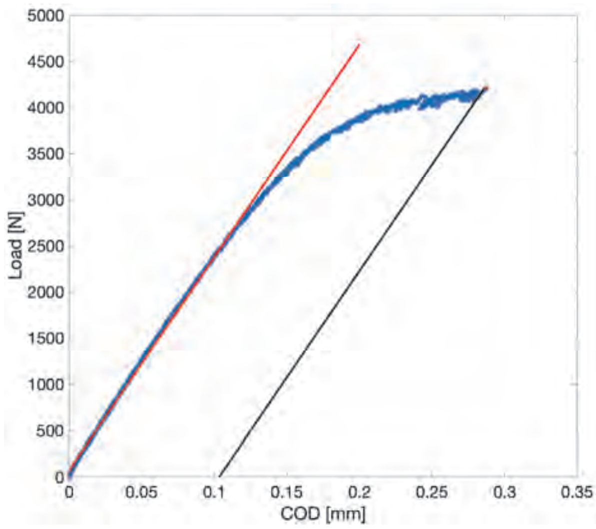

Fig. 4 Dependence of load on COD plot of 15142 steel
Obr. 4 Závislost síly na COD u oceli 15142

Values of fracture toughness that are presented in Tab. 2 do not have too many differences. It can be said that they are similar. The value, which takes into account only elastic properties on materials does not make it possible to determine, which material has better fracture behavior. Value of fracture toughness K_Q does not allow determination of differences between materials.

As it has been said, the value of *J*-integral (J_Q) taking into account plastic behavior allows determining the differences between tested materials.

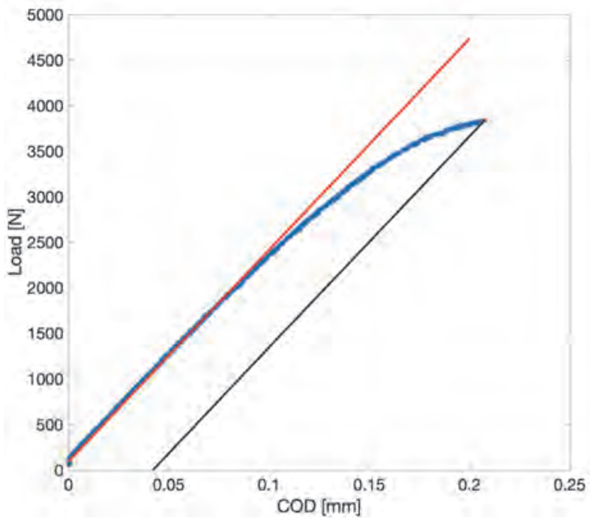

Fig. 5 Dependence of load on COD plot of 14260 steel
Obr. 5 Závislost síly na COD u oceli 14260

Value of *J*-integral mentioned in Tab. 2 showed significant differences in material. Differences can be seen from Figs. 2 – 4.

Each figure defines plot taken from the test. There are plotted *COD* (crack opening displacement) vs. load dependence. Those plots precisely define plastic behavior of tested steels.

On the plots there are marked points for calculating of results, such as a straight line for calculation of P_q and maximal force $P_{max.}$ All calculations were performed by Matlab software.

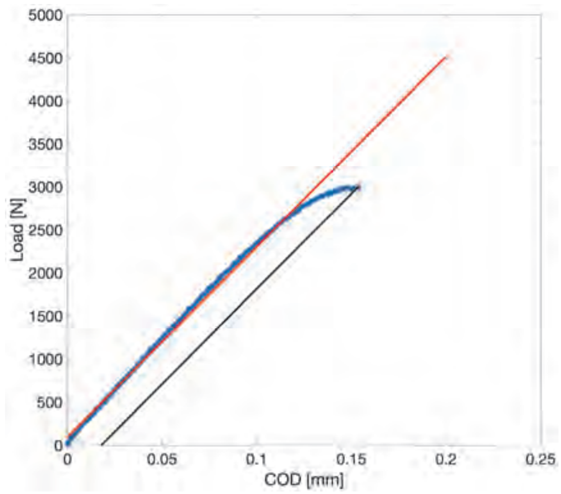

Fig. 6 Dependence of load on COD plot of 1.7765 steel
Obr. 6 Závislost síly na COD u oceli 1.7765

Conclusions

The present work investigated fracture toughness of steels prepared by method of metal injection molding. A study of fracture toughness parameters was conducted on 4 types of those steels. Static fracture toughness tests were carried out with evaluation of K_q and *J*-integral values.

Fractures parameters values obtained using linear elastic fracture mechanics did not give well comparable results. These values have no significant differences between each other, and the fracture properties cannot be evaluated.

Considering results mentioned in table 3 was calculated value of elastic-plastic equivalent K_{JQ} from J- integral as follows:

$$K_{QC} = \sqrt{E' \cdot J_Q}, \tag{7}$$

where

$$E' = \frac{E}{1-\upsilon^2} \tag{8}$$

Comparison of calculated values is shown in table 4.

Tab. 4 Results comparison
Tab. 4 Porovnání výsledků

Steel	K_Q	J_Q **- integral**	K_{JQ}
	(MPa·m$^{1/2}$)	(kJ·m^{-2})	(MPa·m$^{1/2}$)
16 720	69	103	48
15 142	72	44	21
14 260	65	10	15
1.77656	74	20	21

Results obtained by elastic-plastic fracture mechanics (EPFM), i.e. value of *J*-integral (J_Q) and calculated value K_{JQ} seems to be much more suitable for this case. The

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value of J-integral and his elastic-plastic equivalent showed significant differences in fracture properties between the results of individual steels.

By comparison of individual results, it can be seen, that better plastic behavior has material 16720, on the other hand, the steel 1.7765 is brittle material. It means that the material 16 720 has the best resistance to the initiation of an unstable brittle cracking. Based on the results obtained, it can be concluded that the value K_q does not provide a good base for a comparison of the MIM material with high tensile properties. Using the EPFM, we get a good base for comparison of that material because we take into account the minimal plastic property. The results obtained by this method have significant differences.

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References

[1] RICE, J. R. A Path Independent Integral and the Approximate Analysis of Strain Concentration by Notches and Cracks. *Journal of Applied Mechanics* [online], 35 (1968) 2, 379. Available from: <http://appliedmechanics.asmedigitalcollection.asme.org/article.aspx?articleid=1398618>.

[2] ASTM E 1820-17A Standard Test Method for Measurement of Fracture Toughness. In: *ASTM International, West Conshohocken, PA* [online]. 2017. Available from: <www.astm.org>.

[3] PEREIRA, M. V., DARWISH, F. A., CAMPELO, E. On the Relationship between J-integral and Crack Tip Opening Displacement in Elastic-plastic Fracture Mechanics. *Journal of Materials Engineering and Performance*, 22 (2013) 8, 2271–2276.

[4] FARAHMAND, B. Elastic-Plastic Fracture Mechanics (EPFM) and Applications. *Fracture Mechanics of Metals, Composites, Welds, and Bolted Joints*. Boston, MA: Springer, 2001, pp. 180–236. ISBN 978-1-4615-1585-2.

[5] OHJI, T., MRITYUNJAY, S., HALBIG, M., MATHUR, S. E. *Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials VI*. Hoboken, New Jersey: John Wiley & Sons, 2012. ISBN 9781118217528.

[6] TORRALBA, J. M. Metal Injection Molding (MIM) of Stainless Steel. *Handbook of Metal Injection Molding*, Woodhead Publishing, 2012, pp. 393–414. ISBN 9780857096234.

[7] ERDEM, O., URTEKIN, L.: An Investigation of Moldability and Mechanical Properties of MgO Added Alumina. *Kovove Materialy*, 56 (2018) 1, 41–53.

[8] ASTM E8/E8M-16A Standard Test Methods for Tension Testing of Metallic Materials. In *ASTM International, West Conshohocken, PA*. 2016.

[9] ASTM E399-17 Standard Test Method for Linear-Elastic PLane-Strain Fracture Toughness KIc of Metallic Materials. In *ASTM International, West Conshohocken, PA* [online]. 2017. Available from: <www.astm.org>.

[10] ANDERSON, T. L. *Fracture Mechanics: Fundamentals and Applications*. [s.l.]: Taylor and Francais, N.W. Boca Raton, Florida, USA, 2005, 610 pp. ISBN 9781420058215.

Nový postup na obráběcím stroji s aditivní výrobní technologií

Frankfurt nad Mohanem, listopad 2018. Nový postup pro extrémní vysokorychlostní navařování laserem (Ehla) má skutečně úspěšný příběh: od roku 2012 intenzivní vývoj v Cáchách, důsledná realizace a odzkoušení systémové techniky v Nizozemsku a potom nasazení v průmyslu v Číně. Postup, který byl vyvinut ve Fraunhoferově ústavu pro laserovou techniku ILT a na katedře digitální aditivní výroby DAP Univerzity RWTH v Cáchách, obdržel cenu Bertholda Leibingera za inovace. Cena se uděluje od roku 2000 za mimořádné výzkumné a vývojové práce v oblasti využití nebo výroby laserového světla každé dva roky.

Návod na výrobu pravděpodobně nejrychlejšího zařízení pro povrchovou úpravu pomocí laseru je svým způsobem velmi jednoduchý: Vezmeme CNC soustruh, nainstaluje se zdroj laserových paprsků, obráběcí hlava a systém na přivádění prášku – a hotovo. Toto chytré využívání aditivní výrobní technologie bude představeno veřejnosti na veletrhu EMO Hannover 2019.

Nový postup řeší problém, který se týká zvláště výrobců velmi namáhaných kovových dílů. Díly se musí povrchově upravit, aby nedocházelo ke korozi nebo opotřebení. Extrémně vysoké jsou například požadavky na povrchovou úpravu dlouhých válců, které vzhledem ke slanému prostředí v moři rychle rezaví a opotřebovávají se. Tradiční postupy povrchové úpravy, jako je tvrdé chromování, termické stříkání a navařování jsou spojené s nevýhodami. Také laserové navařování se dosud prosadilo v této oblasti jen v jednotlivých případech.

Za úspěch na trhu vděčí vývojáři z Cách také dvěma odvážným holandským průkopníkům. Při hledání výrobce zařízení narazili vědci na zatím mladou firmu Hornet Laser Cladding B. V. z Lexmondu v Nizozemsku, k jejímž zakladatelům Jelmeru Brugmanovi a Franku Rijsdijkovi mají už léta úzkou vazbu. V roce 2014 vzniklo v jejich továrně první zařízení Ehla. Přitom se jednalo v podstatě o „dostrojený“ automatický soustruh. Výhodou povlakování rotačně symetrických dílů je, že lze potřebné komponenty – tedy zdroje laserového paprsku, obráběcí hlavy Ehla a systém přívodu prášku – dobře zaintegrovat a produktivita a systémová technika ve skupině „povlakování laserem“ ve Fraunhoferově ústavu ILT.

- z tiskové zprávy -