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PREPARATION OF COMPOSITE STEEL WITH USING OF HOT ROLLING

PRZYGOTOWYWANIE KOMPOZYTU STALOWEGO METODĄ WALCOWANIA NA GORĄCO

The paper deals with the unconventional issue of composite steel preparation by hot rolling. Various production possibilities of multilayer composite from austenitic stainless steel AISI 304 and tool high carbon steel AISI D2 were investigated. Samples with 5, 15 and 45 layers were prepared. Optical microstructural analysis were subsequently performed on these samples with the focus on the details of joint and evaluation of the thicknesses of individual layers. In addition, hardness measurements were performed on the prepared samples, including its comparison with the individual steels used. It was confirmed that the hardness increased with increasing number of layers, however in this case, the difference between 15 and 45 layers was only 8 HBW. In addition it was found that the hardness of the prepared 45-layer packet without quenching was higher than that of the base (used) steels.

Keywords: *tool steel, stainless steel, composite steel, rolling, metallography, hardness tests*

Niniejszy artykuł poświęcono niekonwencjonalnemu zagadnieniu przygotowania kompozytu stalowego metodą walcowania na gorąco. Zbadano różne możliwości i metody wytwarzania wielowarstwowego kompozytu stalowego z austenitycznej stali nierdzewnej AISI 304 i wysokowęglowej stali narzędziowej AISI D2. W pracy opisano technologię przygotowania pakietów z tych stali złożonych z 5, 15 i 45 warstw. Następnie przeprowadzono na tych próbkach optyczne analizy mikrostrukturalne, skupiając się na szczegółach łączenia i ocenie grubości poszczególnych warstw. Ponadto na przygotowanych próbkach wykonano pomiary twardości, a wyniki porównano z poszczególnymi zastosowanymi stalami. Potwierdzono, że twardość wzrastała wraz ze wzrostem liczby warstw, jednak w przypadku różnicy między 15 a 45 warstwami różnica twardości to zaledwie 8 HBW. Ponadto stwierdzono, że twardość przygotowanego 45-warstwowego pakietu bez hartowania była wyższa niż (użytych) stali podstawowych.

Słowa kluczowe: *stal narzędziowa, stal nierdzewna, kompozyt stalowy, walcowanie, metalografia, badania twardości*

1. INTRODUCTION

Forge welding, or the creation of composite steels, is a technology which, although very old, is still up-to-date and can be used to produce both objects of a decorative nature (for example, knives, swords, rings made of Damascus steel) and objects for practical use (for example, machining tools, tools or kitchen utensils) [1–3]. The basic principle and meaning is the preparation of a product or semi-finished product with a wide range, often even contradictory properties, which are given by a combination of properties of typically different steels [4–6].

The main goal of this work was to design and verify the unconventional procedure for the preparation of composite steel flat blanks. Specifically, it involved the use of longitudinal rolling technology in a reversing duo rougher, which is part of the laboratory semi-continuous rolling mill at VŠB-TU Ostrava. To implement this experiment, two types of very different steels were used (austenitic stainless steel AISI 304 and high-carbon tool steel AISI D2). 5-, 15- and 45-layer composite blanks (packets) were prepared. Subsequently, the quality of joints between the individual layers was assessed on the basis of metallographic analysis. Finally, the effect of the number of layers on hardness was also assessed, and the hardness results were also compared with the hardness values of the base steels.

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2. EXPERIMENT DESCRIPTION

The reversible duo rougher (two-high rolling mill) that was used to carry out the experiments is presented in Fig. 1. It is the first part of the semi-continuous laboratory bar rolling mill. The next (continuous) part is formed by four two-high rolling mills in tandem layout arranged one behind the other in the HVHV configuration (H – horizontal and V – vertical placement of the stand). This rolling mill is primarily intended for simulations and optimization of temperature-controlled rolling and cooling processes of round bars, as well as for studying intensive hot forming processes [7].

As the goal was to create flat semi-finished products, only flat parts of the rolls with a flat oval-circle calibration (Fig. 1b) were used. Mobile electric resistance furnaces were used to heat the packets.

The experiment was realized with the aim of combining two steels fundamentally different in type and chemical composition, and thereby achieving a combination and compromise in their functional properties. Specifically, it was tool steel AISI D2 (DIN 1.2379) and austenitic stainless steel AISI 304 (DIN 1.4301), and their chemical composition is described in Table 1 [8]. The AISI 304 steel is used in engineering, water supply, gastronomy and healthcare. It is the most widespread stainless steel on the market with relatively good formability, weldability and also good cold ductility [9]. Tool steel AISI D2 is designed for cold work. It has high abrasion resistance, good hardenability and higher corrosion resistance. It is used for cutting and pressing tools, industrial knives and punches [10, 11].

Sheets with a thickness of 3 mm, a width of 50 mm and a length of 120 mm were used from both mentioned steels. The cut sheets were sanded, cleaned, degreased and finally stacked alternately into 5-layer packets (three AISI 304 steel layers and two AISI D2 steel layers). Then, they were welded around the entire perimeter so that the cohesion of the materials in the longitudinal and vertical axis of the packet was maintained during rolling. The welding was done by the MMA method (manual electric arc welding with melting electrodes). 15-layer packets were welded from three rolled 5-layer packets and 45-layer packets from three rolled 15-layer packets. In Fig. 2, a photo of the already made five-layer package is presented. After the rolling of all three variants, the samples were subjected to metallographic analysis (optical microscopy) with the focus on measuring the thickness of individual layers and evaluation of joints between layers. HBW hardness was measured as well.

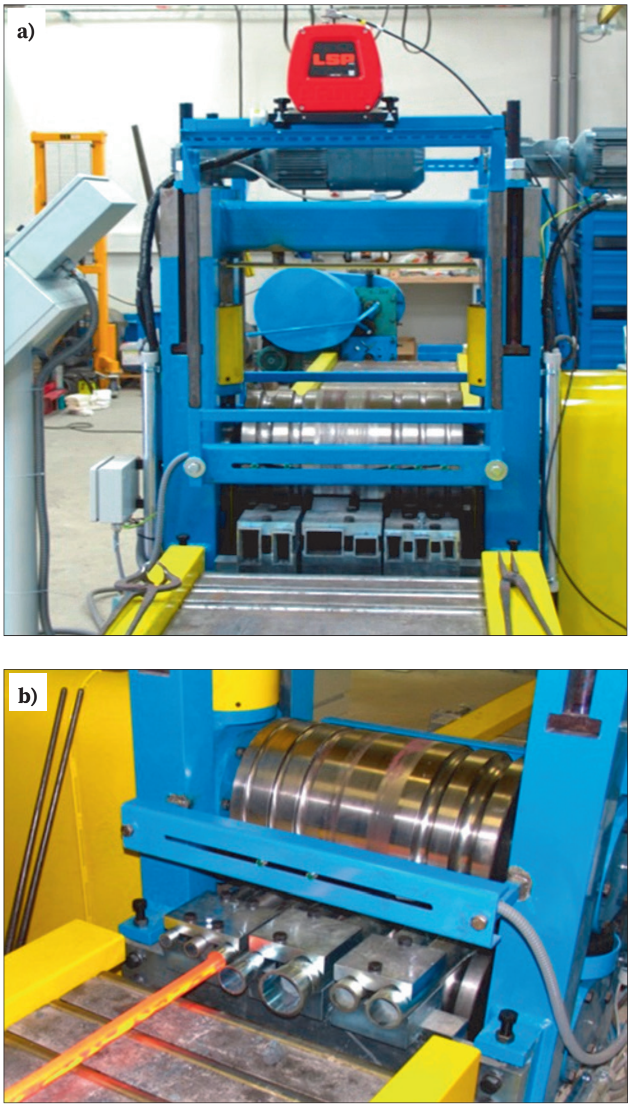

Fig. 1. Reversing duo rougher [7]

Rys. 1. Odwracalna walcarka podwójna [7]

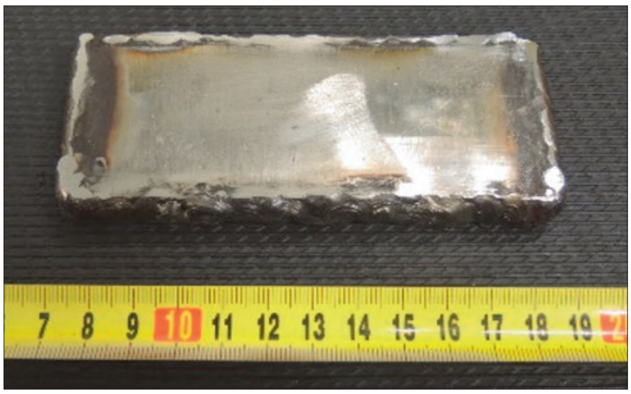

Fig. 2. Five-layer packet, ready to roll

Rys. 2. Pakiet pięciowarstwowy, gotowy do zrolowania

Table 1. Chemical composition of used steels in wt. % [8]

Tabela 1. Skład chemiczny stosowanych stali w % wag. % [8]

Steel	C	Si	Mn	Cr	Ni	Mo	V	P	S
AISI D2	1.460	0.258	0.239	11.2	0.197	0.769	0.711	–	–
AISI 304	0.08	1	2	18	8	–	–	0.04	0.03

3. ROLLING PROCEDURE AND RESULTS

At the beginning of the experiments, an important negative influencing factor was found, namely due to the imperfect grinding of the passive layers (difficult to remove especially from the AISI D2 steel sheets), pressure welding of the materials did not occur due to rolling, or their delamination occurred. In the Fig. 3 it is visible that one of the layers of the test sample has separated from the others. For this reason, new samples (packets) were produced, which were already assembled with more thoroughly prepared surfaces, which did not show this fundamental deficiency after rolling.

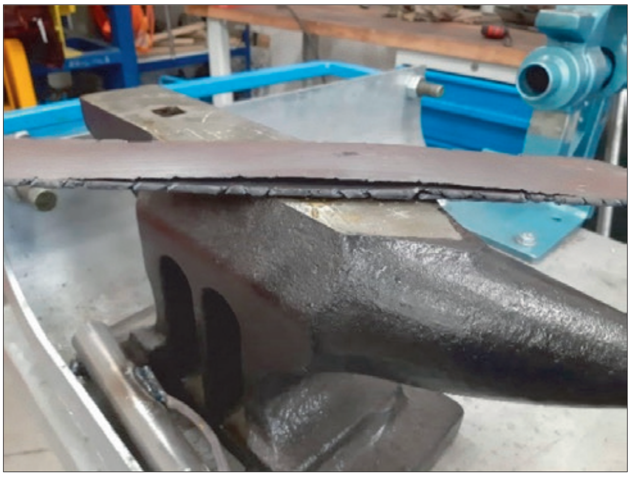

Fig. 3. Delaminated sample

Rys. 3. Delaminowana próbka

So in the next stage, three 5-layer packets were first assembled, with a total thickness of 13.6 mm, which means that each input sheet was ground on each side by approximately 0.14 mm. The packets prepared and welded in this way were subsequently rolled in seven passes to a final thickness of 4.3 mm – measured while hot (4.2 mm when measured while cold). The heating temperature was 1100°C with the 20 minute holding time in the oven. The final thickness of 4.2 mm was chosen due to the possibility of metallographic analyses and at the same time to obtain a semi-finished product suitable for possible further processing with the aim of producing a functional sample of the knife. Furthermore, the first, third and fifth rolling pass was followed by interpass heating again to a temperature of 1100 °C with 5 minute holding time.

Since in previous experiments, which were not part of this work, it was verified that this rolling mill allows obtaining up to 50% reduction in height in the first pass under the selected heating conditions of the investigated composite, therefore, with the assumption of achieving a high-quality joint, this reduction was ~~used~~ selected. However, due to the effect of roll jump the resulting reduction in height in the first pass was slightly smaller, about 45%. The values of ΔH – absolute reduction in height, ε_H – engineering strain and e_H – true strain in the subsequent passes are presented in Table 2. Passes 3-7 was carried out in all cases with the same setting of the rolling gap (4 mm) and the deformation was thus influenced only by the jump of the rolls.

Table 2. Dimensions before and after rolling and strain values

Tabela 2. Wymiary przed i po walcowaniu oraz wartości odkształceń

	NL5				NL15 and NL45			
	H	ΔH	εH	eH	H	ΔH	εH	eH
	(mm)	(mm)	(%)	(-)	(mm)	(mm)	(%)	(-)
0	13.6	–	–	–	12.6	–	–	–
1	7.6	6	44.1	-0.58	6.9	5.7	45.2	-0.60
2	6	1.6	21.1	-0.24	6.25	0.65	9.4	-0.10
3	5.05	0.95	15.8	-0.17	5.25	1	16.0	-0.17
4	4.6	0.45	8.9	-0.09	4.7	0.55	10.5	-0.11
5	4.45	0.15	3.3	-0.03	4.45	0.25	5.3	-0.06
6	4.4	0.05	1.1	-0.01	4.4	0.05	1.1	-0.01
7	4.3	0.1	2.3	-0.02	4.3	0.1	2.3	-0.02
Total	–	9.3	68.4	-1.151	–	8.3	65.9	-1.075

In the case of 15-layer packets, two such packets with a thickness of 12.6 mm were prepared by cutting all 5-layer packets in half (a small part was left from the central part for metallographic analysis).

Only one packet with 45 layers was prepared, while the previous procedure was again used, i.e. 15 layer packets were divided into halves, keeping the central part of the sample for subsequent metallographic analysis. The 45-layer packet was thus composed of three surface-ground scraps from 15-layer rolled packets and had a height of 12.6 mm. After finishing the rolling, the packets were cooled freely in the air. In Fig. 4–6, the courses of rolling torques, rolling forces and setting of the rolling gap are recorded as a function of time. Markings NL5, NL15, NL45 then represent the sample designation (NL – Number of Layers) and its number of layers (5, 15, 45).

Fig. 4 shows the rolling data registered for sample NL5, which, as expected, had the lowest flow stresses of all three sample variants during rolling, and respectively the lowest measured rolling forces and torques during all passes. The largest rolling force of approx. 561 kN and the largest rolling torque of 17.4 kNm was measured in the first pass.

When rolling a sample with 15 layers, it was immediately apparent that a larger number of layers in the packet has a noticeable effect on the strength properties, respectively on the flow stress. It is only fully comparable in the last graph (Fig. 7), where the last two passes are compared, but it can be assumed already in the first pass, for example, due to the fact that the first pass has a force maximum of 619 kN. In the case of the rolling torque, we move to a maximum

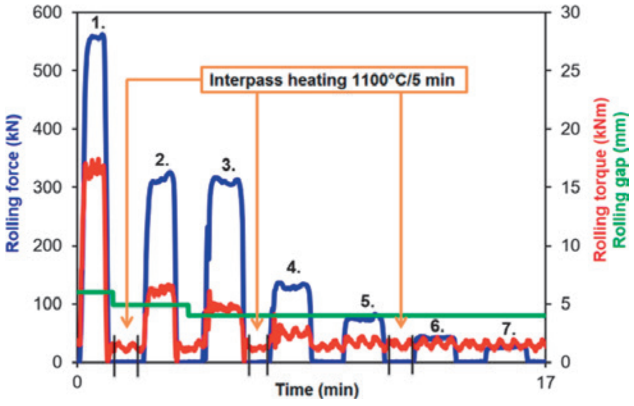

Fig. 4. Graph of the rolling data for the sample NL5
Rys. 4. Wykres danych toczenia dla próbki NL5

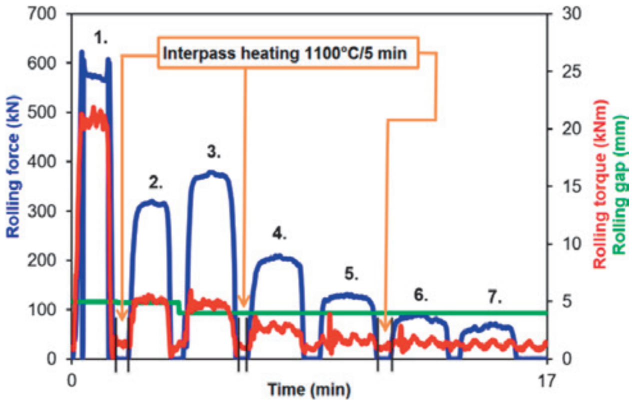

Fig. 5. Graph of the rolling data for the sample NL15
Rys. 5. Wykres danych toczenia dla próbki NL15

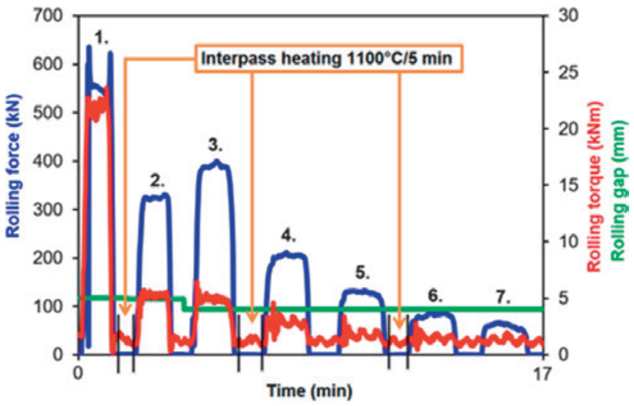

Fig. 6. Graph of the rolling data for the sample NL45
Rys. 6. Wykres danych toczenia dla próbki NL45

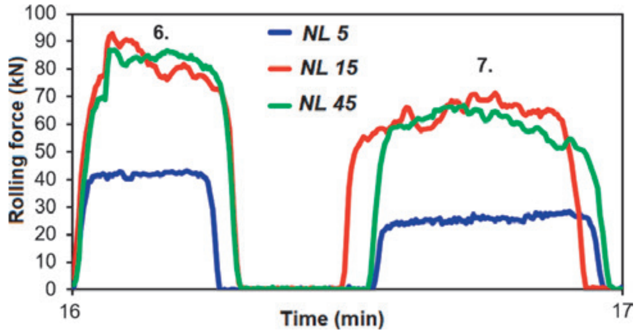

Fig. 7. Comparison of rolling forces for the 6th and 7th pass
Rys. 7. Porównanie sił walcowania dla 6. i 7. przejazdu

of 21.85 kNm for the NL15 sample, which is 4.45 kNm more than in the case of the NL5 sample. The higher energy requirements for the rolling mill and its power mechanism can therefore be confirmed.

From the graphs in Figs. 5 and 6 it is clear that the differences in rolling forces and torques are not so significant for packets with 45 and 15 layers. The maximum rolling force for the sample NL45 in the first pass is 632 kN, which is 12 kN more than in the case of sample NL15, but when comparing the last two passes, the values are almost comparable. The maximum rolling torque for the sample NL45 in the first pass is 23.62 kNm, which is 1.77 kNm more than in the case of sample NL15.

When comparing the rolling forces in the last two passes (when the passes plan is completely identical) for all three samples (Fig. 7), it can be stated that as the number of layers increases, the deformation resistance also increases significantly, but only in the range of the number of layers 5–15.

4. METALLOGRAPHIC ANALYSIS AND DISCUSSION OF RESULTS

This experiment also included subjecting the samples to optical metallographic analyses. On three samples (NL5, NL15 and NL45) a transverse section was made, which is perpendicular to the longitudinal axes of the samples, and metallography was made in the middle of their width and length. Photo documentation of the macrostructure using a scanner was first performed, as can be seen in Fig. 8.

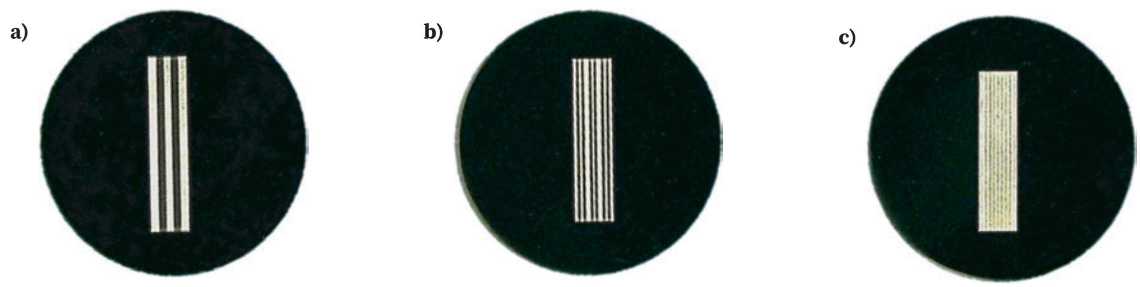

Fig. 8. Macro images of the tested samples: a) NL5, b) NL15, c) NL45
Rys. 8. Zdjęcia makro badanych próbek: a) NL5, b) NL15, c) NL45

To improve the visibility of the macrostructure, the samples were etched with chemical etching solution L-1 based on ferric chloride. After etching, the given layers can already be observed. For all samples, the light layers represent AISI 304 austenitic stainless steel and the dark layers represent AISI D2 tool steel. No defects or signs of non-bonding (delamination) were recorded in all metallographic images (both macro images and micro images). The microstructure of the samples was photo-documented mainly with the aim of focusing on the greatest possible details of joint and changes in the structure in this area – see in Fig. 9–11.

In the samples with 5 and 15 layers (Figs. 9 and 10), the joint between the layers was compact despite the fact that there were small chains of iron oxides in the joint. Despite this phenomenon, the materials can be considered compact and the quality of the joint is good. Furthermore, it is clear that with the increasing number of layers, the grain becomes finer (mainly at the junction of the layers) and there is also an undesirable effect of decarburization of AISI D2 tool steel. At the same time, the purity of the joint itself seems to increase with more layers. The decarburization of AISI D2 steel is apparently the result of repeated heating of the steel to a temperature of 1100 °C, which is the limiting forming temperature of of this steel. Overall, AISI 304 steel has a finer grain.

In the case of sample NL45, the joint of two identical layers (red border area) of AISI 304 steel was also documented (Fig. 12). The joint is again in some places lined with small chains of probably iron oxides, and in other places it is perfectly clean with an imperceptible transition between individual layers.

The evaluation of the metallographic images was followed by the thickness measurement of the individual layers for all three samples. The thicknesses were measured and photo-documented at the same time (Fig. 9–11) using the Quick PHOTO INDUSTRIAL 3.2 software and the values were recorded in Table 3, which is also supplemented with the measured HBW hardness values.

The graph in Fig. 13 shows the relationship between the average layer thickness, the HBW hardness and the total number of layers in the sample. As the thickness of the individual layers decreased, the

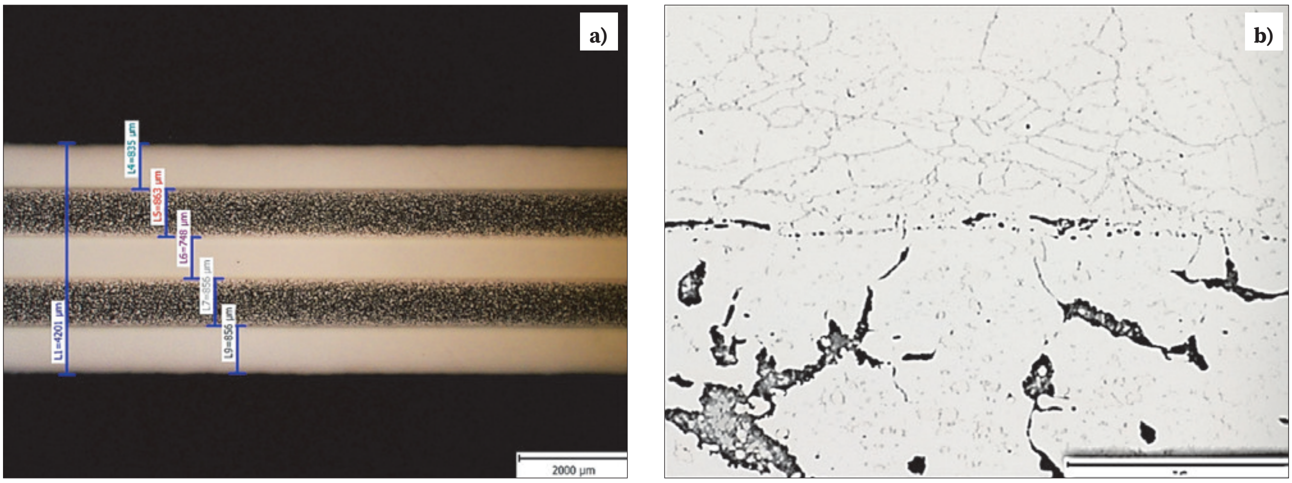

Fig. 9. Metallographic images of sample NL5, a) magn. 12.5×, b) magn. 1000×

Rys. 9. Zdjęcia metalograficzne próbki NL5, a) powiększenie 12,5×, b) pow. 1000×

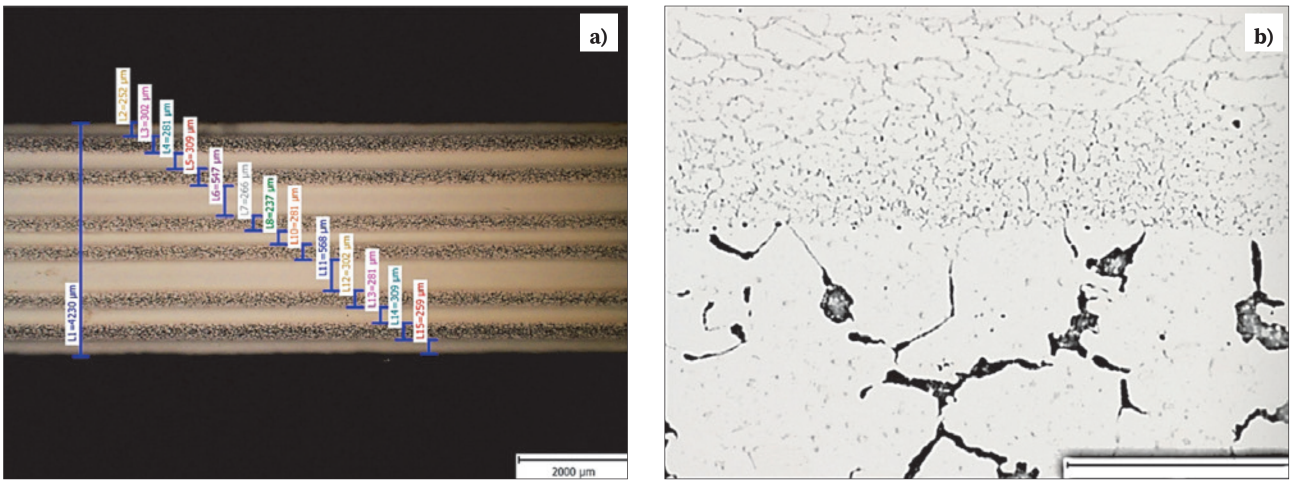

Fig. 10. Metallographic images of sample NL15, a) magn. 12.5×, b) magn. 1000×

Rys. 10. Zdjęcia metalograficzne próbki NL15, a) powiększenie 12,5×, b) pow. 1000×

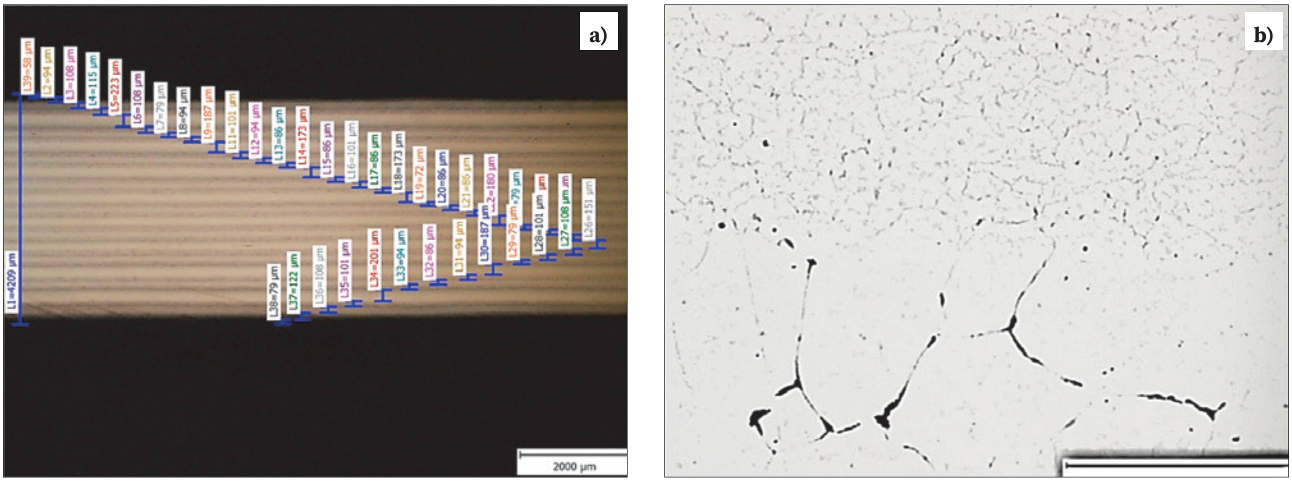

Fig. 11. Metallographic images of sample NL45, a) magn. 12.5×, b) magn. 1000×

Rys. 11. Zdjęcia metalograficzne próbki NL45, a) powiększenie 12,5×, b) pow. 1000×

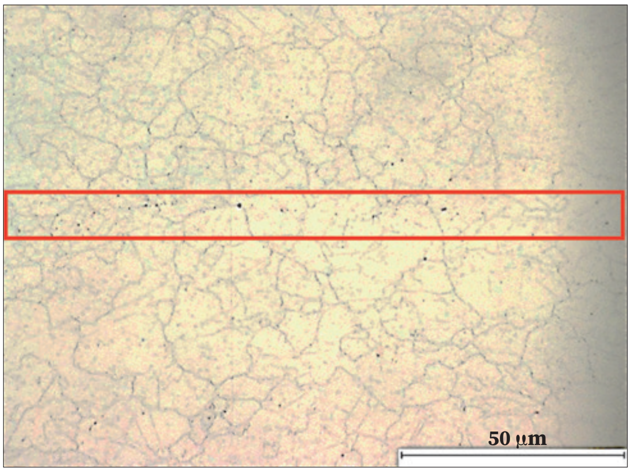

Fig. 12. A metallographic image of the joint of two AISI 304 steel layers in the sample NL45, magn. 1000×

Rys. 12. Metalograficzny obraz połączenia dwóch warstw stali AISI 304 w próbce NL45, pow. 1000×

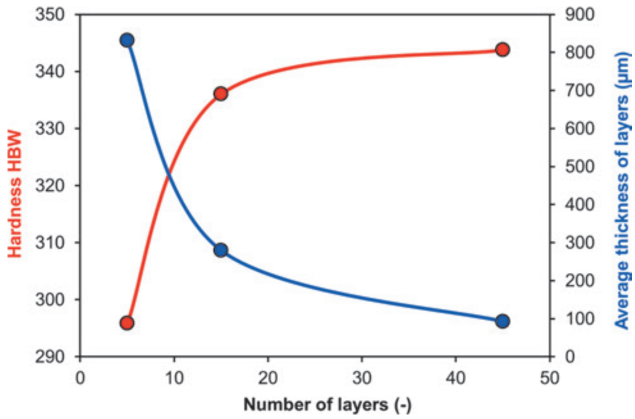

Fig. 13. Dependence of hardness and average thickness of layers on the number of layers

Rys. 13. Zależność twardości i średniej grubości warstw od liczby warstw

standard deviation of the individual layer thickness also decreased, of course. However, from the measurement of the thicknesses of the individual layers, it is primarily that the layers of both joined steels had the same thickness and therefore the same deformation. This positive fact was most likely ensured by the all-round weld. In the case of using only spot welds, which are often applied in these welding damascus production procedures, the situation would probably be different and the less strong and more malleable stainless steel would probably elongate more and therefore it would have a smaller layer thickness.

In the graph (Fig. 13), it can be noticed that with the increasing number of steel layers in the packet, the hardness of the samples also increased. The 5-layer sample has the lowest value, starting at 296 HBW. The following sample NL15 has a much higher HBW hardness value, namely 336 HBW. It is clear from the graph that the difference in hardness between the 5-layers packet and the 15-layers one is far greater (benefit for 15-layers packet) than between the 45-layers and the 15-layers packets. It is therefore possible that the production of this particular composite steel, which exceeds 45 layers, is ineffective

Table 3. Data from layer thickness and hardness measurements

Tabela 3. Dane z pomiarów grubości i twardości warstwy

Sample	Average thickness of layers (µm)	Total thickness of samples (µm)	Standard deviation (µm)	Hardness HBW
NL5	831.60	4201	42.8	296
NL15	279.63	4230	20.1	336
NL45	92.78	4209	12.4	344

for increasing hardness, and apparently, the other mechanical properties. It should be added that in all three cases the test sample or the material from which it was taken was not heat-treated (or hardened) in any way, which of course could fundamentally affect the hardness measurement results.

Table 4 shows a comparison from which it follows that the preparation of a composite steel material of the given composition brings an increase in the hardness of the sheet. However, as already mentioned above, the ideal limit between the ratio of the achieved hardness and the number of layers in the material for this case is around 45 layers, when this particular composite material approaches its maximum hardness. A maximum hardness of 344 HBW was achieved for the composite steel with the given preparation procedure and composition. For comparison, the maximum hardness value for non-heat-treated AISI 304 steel is added, which is 201 HBW, and for tool steel AISI D2, this value is around 255 HBW [12].

Table 4. Comparison of the maximum achievable hardness for the given materials [12]

Tabela 4. Porównanie maksymalnej osiągalnej twardości dla danych materiałów [12]

Material	Maximum achievable hardness HBW
Composite steel, AISI 304 + AISI D2 (sample NL45)	344
Tool steel AISI D2	255
Stainless steel AISI 304	201

5. CONCLUSIONS

In this article, the issue of preparing specific multi-layer steel composites using rolling technology was described. A procedure for rolling 5, 15 and 45 layer packets of AISI 304 stainless steel and AISI D2 tool steel was successfully designed and tested.

For all three samples, the preparation result can be considered successful, as sufficiently high-quality joints between the layers were achieved and this composite steel showed better mechanical properties, or hardness, than the input steels of which it was composed. Metallographic images were documented and layer thicknesses were measured, proving that the joints between the layers were compact. There was also a slight grain refinement, which again may contribute to improved mechanical properties.

However, these successful results were preceded by unsuccessful attempts to prepare this material, which were mainly caused by problems with the removal of the passive layer in AISI D2 tool steel sheets.

With the specific composition of the material being prepared, it was also proven that, in the case of hardness evaluation without prior hardening or other heat treatment, it no longer makes much sense to increase the number of layers above 45, since there is no significant increase in hardness expected.

The results of this experiment in the form of gained experience and in the form of obtained semi-finished products can be beneficial and can serve for further experiments and further knowledge in this issue.

Acknowledgements

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REFERENCES

[1] G. Löbach. *Damaszenerstahl Theorie und Praxis*. Rosenheimer Straße 22, D-83043 Bad Aibling: Weiland Verlag GmbH, 2009. 173 p.

[2] A.K. Radchenko, T.K. Gogaeva. Multilayer Steel Composite Prepared by Rolling. *Powder Metallurgy and Metal Ceramics,* 2003, 42, pp. 336-340.

[3] T. Kubina, J. Nacházel. Mechanické vlastnosti laminárních kompozitních ocelových materiálů připravených válcováním za tepla. *Kovárenství*, 2015, 54, pp. 29-33.

[4] J. Wadsworth, D.R. Lesuer. Ancient and modern laminated composites – From the Great Pyramid of Gizeh to Y2K. In: *International Metallographic Society 1999 Conference*. California, 14.03.2000.

[5] J. Lau. Low-Strain Warm Pressing of Multilayer Composites Yields Layers with Longer-Range Protection from Crack Deflection. *MRS Bulletin*, 2001, 26 (2), pp. 89.

[6] D.R. Lesuer, C.K. Syn, O.D. Sherby, J. Wadsworth, J.J. Lewandowski, W.H. Hunt. Mechanical behaviour of laminated metal composites. *International Materials Reviews*. 1996, 41 (5), pp. 169-197.

[7] S. Rusz, I. Schindler, P. Kawulok, R. Kawulok, P. Opěla, V. Ševčák. Vybrané aplikační možnosti laboratorní polospojité válcovny tyčí. In: Možnosti studia deformačního chování materiálu a fyzikálních simulací procesů válcování na FMMI VŠB-TUO, *Sborník přednášek z 81. pracovního semináře Společnosti Ocelové pásy*. Ostrava, 2018, pp. 28-44.

[8] A. Razaq, N. Mohammed, M.Z. Omar, S. Al-Zubaidi, K.S. Alhawari, M.A. Abdelgnei. Weldability and Joining Characteristics of AISI D2/AISI 304 Steels Using Semisolid Diffusion Joining. *Solid State Phenomena*, 2019, 285, pp. 115-120.

[9] DIN EN 1.4301 Stainless Steel X5CrNi18-10 Material Equivalent, Properties, Chemical Composition. [Online] Available at: https://www.theworldmaterial.com/din-en-1-4301-material-x5crni18-10-stainless-steel/

[10] Nástrojová ocel. [Online] Available at: http://toolsteel.cz/nastrojova-ocel/

[11] DIN EN 1.2379 Steel X153CrMoV12 Material Equivalent, Datasheet, Properties. [Online] Available at: https://www.theworld-material.com/din-en-1-2379-steel-x153crmov12-material/

[12] D. Pasečný. *Preparation of composite steel*, Diploma Thesis, VŠB-TU Ostrava, 2021.