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SURFACE PRETREATMENT OF SELECTIVELY LASER MELTED 316L STAINLESS STEEL SUBSTRATE AND ITS EFFECT ON THE QUALITY OF THE VITREOUS ENAMEL COATING

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Abstract
This paper examines various surface pretreatments and their impact on the quality of vitreous enamel coating. In this case, 316L stainless steel was chosen as the base material and a premix enamel coating specially prepared for stainless steel for enamelling. Samples prepared by Selective Laser Melting (SLM) were used. Both mechanical (blasting, tumbling) and chemical (pickling) pre-treatments were applied. The surface variation of the differently prepared samples together with the interface between the vitreous enamel coating and the base material were examined by SEM. The results show that different surface pretreatments affect the final enamel coating and its quality.

Keywords:
Vitreous Enamel, Surface Treatment, Coating, SLM, Additive Technology

1 INTRODUCTION

In the realm of advanced manufacturing technologies, Selective Laser Melting (SLM) has emerged as a transformative process, pushing the boundaries of traditional material fabrication [Jandaghi 2021, Saboori 2020]. Which include, among others, forming and welding [Cada 2021, Rusz 2020]. Selective Laser Melting, a powder-bed fusion additive manufacturing process, enables the layer-by-layer deposition of metal powders, providing unprecedented design freedom and material optimization [Mosallanejad 2021, Melia 2020].

The study of enamelling steel substrates produced by SLM represents a captivating exploration into the intersection of additive manufacturing and surface engineering [Kanyak 2019, Mahmood 2022, Maleki 2021]. Enamelling, a centuries-old technique traditionally applied to ceramics and metals, is now undergoing a change of concept thanks to the integration of SLM, opening new avenues for enhanced functionality, durability, and aesthetic appeal.

As steel substrates play a pivotal role in various industrial applications [Tatickova 2023], from automotive components to consumer electronics, the ability to enamel such substrates through SLM introduces a novel dimension to their utility. This study delves into the intricate relationship between the unique microstructural characteristics imparted by SLM and the subsequent enamelling process, exploring the synergistic effects that arise from this convergence.

This study focuses on bridging the knowledge gap between two seemingly disparate fields - additive manufacturing and traditional enamelling techniques [Sternadelova 2023]. Through a multidisciplinary approach, we seek to elucidate the complexities associated with enamelling steel substrates produced by SLM technology [Sternadelova 2021], to encourage innovation and push the boundaries of what can be achieved in the ever-evolving landscape of materials science and manufacturing technologies.

2 EXPERIMENTAL

2.1 Sample preparation

The samples used for the experiment were 316L stainless steel samples, which were produced using additive SLM technology, on a Renishaw AM 400 production machine. The dimensions of the samples were (50 x 50 x 5) mm.

The printed samples are significantly influenced by the input material itself, which is specified by ASTM International 52900 standard [ASTM 52900:2015]. The input metal powder was produced by the gas atomization method [Bukovec 2018]. A non-magnetic, austenitic, corrosion-resistant steel with a very low percentage of carbon, which is alloyed with chromium, nickel, molybdenum, and some other elements in minority.

2.2 Specification of sample pretreatment

After printing and removing the supports, the samples were degreased. Degreasing was carried out in isopropyl alcohol in an ultrasonic cleaner at 60 °C for 20 min. The samples were divided into five groups and variously pretreated. For an easier orientation, the sets of samples were labelled with individual letters indicating the types of pretreatments. The

order of the samples is as follows: the first samples (R) are only degreased. P samples are blasted with Polgrit abrasive with a grain size of 0.2-0.5 mm. Samples K are blasted with 90 µm grain white corundum. O samples are tumbled in a centrifugal tumbler for 6 hours. M samples are pickled in 10 % hydrochloric acid (HCl) with hexamethylentetraamine (Urotropin) as an inhibitor, followed by neutralization with 10 % sodium hydroxide (NaOH).

R – samples in original condition were only degreased and the enamel coating was applied to the raw surface of the as-printed stainless steel samples. Usage of as-printed samples was included in the experiment because of possible considerable savings in time and resources for pre-treatment prior to coating application.

P – samples were blasted with Polgrit in this set of samples. Polgrit is a copper slag produced as a by-product of copper extraction by smelting. The slag is rapidly cooled with water and then solidified into coarse, sharp granules. The abrasive is amorphous, chemically neutral, does not react with the work surface and does not cause secondary corrosion. Polgrit abrasive is non-flammable, non-hygroscopic, non-magnetic, non-conductive and, due to its low content of free SiO2, hygienically safe. Furthermore, it does not cause serious health hazards or contribute to environmental pollution. Therefore, its use makes both ecological and economic sense.

The samples from this set were blasted according to EN ISO 8501-1 standard [ISO 8501-1:2007] and determined to be of Sa 3 purity, i.e., very thorough blasting.

K – samples were blasted with white corundum. White corundum was chosen for its most common use in practice. It is a fine alumina, obtained synthetically by melting Al2O3 in an arc furnace. Compared to conventional alumina, it is more homogeneous in terms of chemical composition, structure, and properties. Synthetic white corundum is an environmentally clean and very hard material, resistant to aggressive chemical factors.

O – tumbled samples: To mitigate the surface roughness, a mechanical tumbling pre-treatment method was used in a centrifugal tumbler. For the SLM method, plastering is widely used as a finishing operation, especially for shape complex components where the tumbling media reaches the interior spaces. Furthermore, this technology can be used to remove burrs, round edges, smooth and polish the surface. The entire set of samples was tumbled in a centrifugal disc tumbler (CF1-32 EL, OTEC) for 6 hours.

The tumbling process was divided into 3 2-hour sections. In each section, different tumbling bodies were used in the following order: ceramic DZS 10/10 (OTEC), plastic XS 12K (Walther Trowal) and porcelain P 2/5 (OTEC). During the overall tumbling process, the KFL tumbling compound (Walther Trowal) was used.

M – pickled samples: As another type of pretreatment, chemical pretreatment was chosen, namely pickling at room temperature, in 18 % solution of HCl and Urotropin, for 18 min. Pickling removes scale or corrosion layers, but also dissolves the metal matrix, which can cause hydrogen diffusion into the metal matrix. Therefore, the inhibitor Urotropin, was added to the acid to promote dissolution of oxides without simultaneously increasing metal dissolution. So-called pickling inhibitors reduce the effect of the pickling acid on the pickled base material, preventing the incorporation of hydrogen into the metal lattice and the formation of hydrogen embrittlement. In this case pickling was used to reduce surface roughness. To stop the chemical reactions on the surface of the samples, a 10 % NaOH solution was used for 5 min. Subsequently, the samples were rinsed with tap water, cleaned with isopropyl alcohol, and dried.

2.3 Enameling process

Before the enamelling process, the individual samples were again cleaned with isopropyl alcohol in an ultrasonic cleaner for 5 minutes. They were then pulled out and dried with compressed air.

This was followed by the enamelling process, which in all cases was carried out by dipping the samples. Each sample was dipped. It was pulled out and placed in the oven for 9 minutes at 100°C. A suspension of enamel premix (Wendel Email) for stainless steel and distilled water was used for enamelling. The density of the suspension was 1.7 g·ml-1 as recommended by the manufacturer.

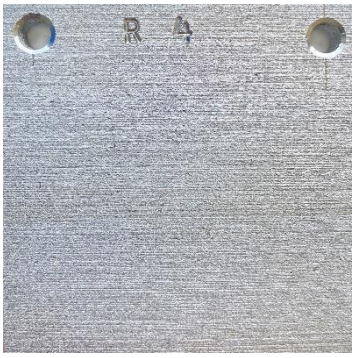
The drying process was followed by a firing process in an oven at 810°C for 5 minutes.

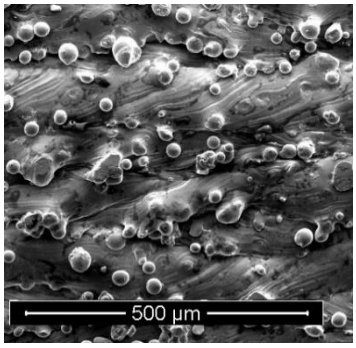
3 RESULTS

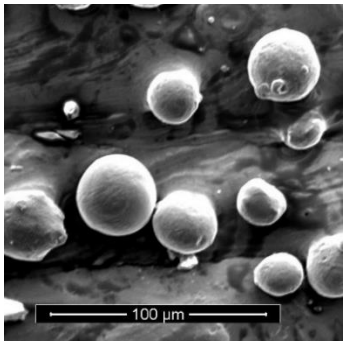
3.1 Surface analysis

The surface topography of all samples was examined using a scanning electron microscope (SEM, FEI Quanta 450FEG). The images were taken in secondary electron (SE) detection mode and an accelerating voltage of 15 keV.

R







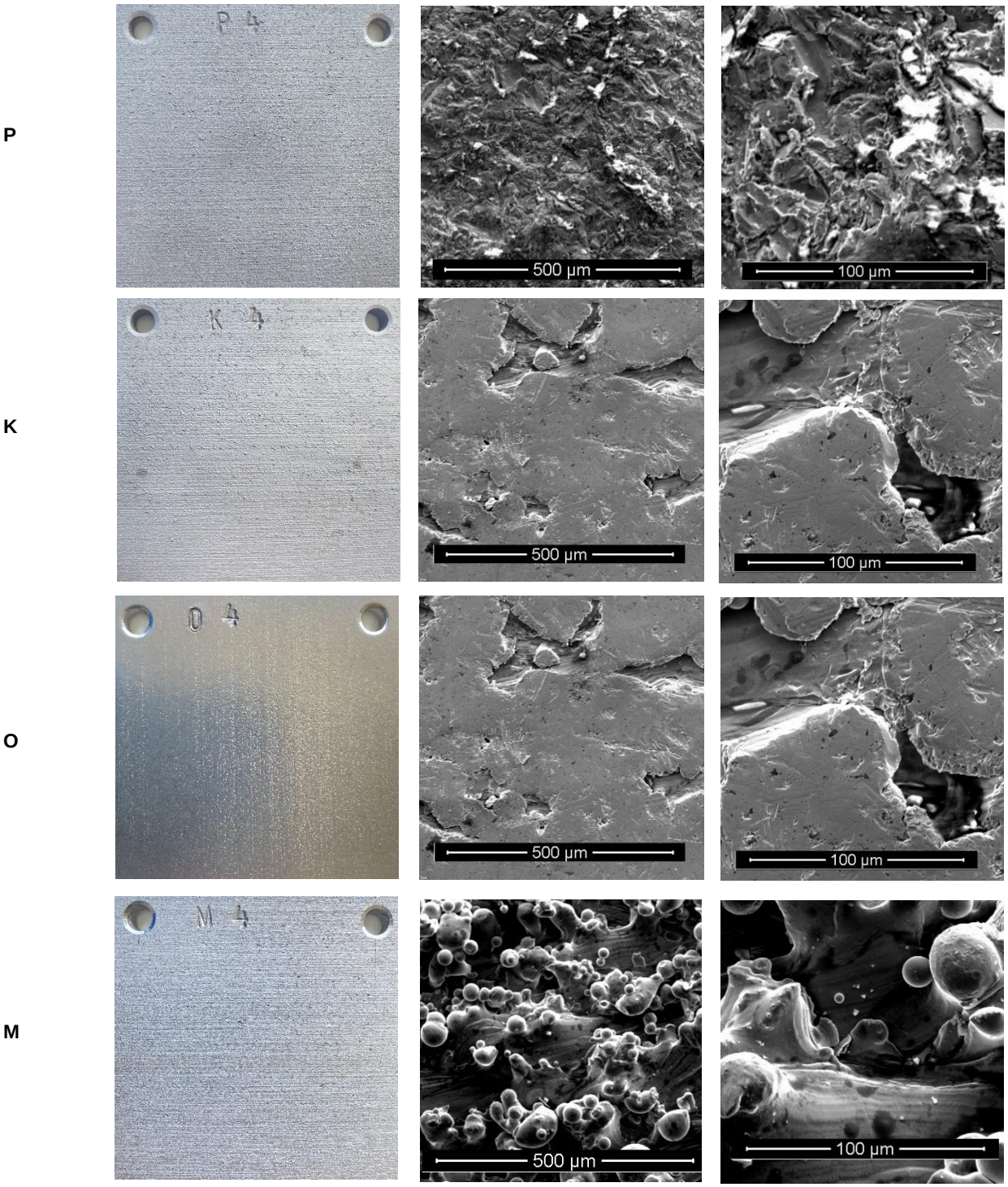


Fig. 1: Comparison of all types of sample pretreatments. From left to right respectively: real sample image, SEM image with 200x magnification and SEM image with 500x magnification.

The figure contains a comparison of all types of sample pretreatments. From left to right respectively: real sample image, SEM image with 200x magnification and SEM image with 500x magnification.

The images of sample R show the standard structure after printing. In sample P, the change in structure due to mechanical pre-treatment by blasting can be seen and the enlarged images show the contamination caused by imperfect cleaning of the abrasive. The structure of sample K (white corundum blasted) is different from the two previous structures. It can be seen that the surface is uniform, which generally improves the adhesion of the coatings.

The O samples (tumbled) have a smooth surface with visible defects caused by additive manufacturing. Such a smooth surface may have a negative effect on the adhesion of the coating or on the application of the enamel suspension. Due to the small anchoring profile, it is possible that the suspension does not adhere sufficiently to the sample surface. Tumbling is the most common finishing operation in additive manufacturing; therefore, this pretreatment method has been included. The M samples were chemically pretreated. The surface of the samples so treated is very different from the previous mechanically pretreated samples. The chemical pre-treatment of the surface by pickling caused considerable porosity of the surface, but it can be seen that the pickling was carried out for a sufficient time and the surface was pickled uniformly.

3.2 Roughness measurements

Roughness measurements were performed on the InfiniteFocus measuring system (Alicona) using the non-contact Focus Variation method described in EN ISO 25178-606 standard [ISO 25178-606:2016].

Among the selected parameters, the roughness height profile parameters were chosen. The parameter Ra is statistically very stable and repeatable, but it cannot respond sufficiently to local height differences in the profile of the surface under study. The newly created product using the additive manufacturing has a specific surface. This is created by laser melting a special metal powder, poured in layers, which melts to form a solid mass. During printing, this creates pores in the material that are part of the surface. Therefore, the additional height parameters of the roughness profile Rz, Rp and Rv were chosen to have an optimal predictive power for the surfaces we investigated, as they are sufficiently responsive to local height irregularities on the profile under evaluation.

Similar to profile height parameters, area height parameters have a similar application. Area analysis is performed on a defined area of the sample and can capture the highest peak and deepest bottom of the surface texture, unlike profile analysis, which works with measured line data. The planar parameters are determined from a much larger amount of measured data, which gives us a more objective presentation of the area inspected. Calculated areal parameters have a more accurate predictive power. It is not always possible to measure areal parameters, and profile roughness parameters are still used in practice to determine surface quality. Profile and surface parameters are not equal, but their comparison is possible. For these reasons, both profile and surface roughness parameters were selected for the quantified description of the topography of the samples examined, and their values can be compared, as well as the functional parameters Spd and Spc.

During the measurements, a (5 x 5) mm area was scanned on the samples. 5 different surface topographies were measured after three repetitions.

Table 1 shows the measured mean values of the profile and surface roughness parameters. According to these values, it can be concluded that the surface was the roughest in case of the raw R samples, while the least rough surface was found in the tumbled O samples.

Tab. 1: Roughness – measured mean values

Roughness (µm)	R	P	K	O	M
Ra	10.90	7.54	7.25	0.44	8.03
Rz	110.92	46.82	51.24	4.71	57.95
Rv	73.43	22.62	26.77	3.94	28.15
Rp	37.49	24.20	24.47	0.77	29.80
Sa	11.60	8.29	7.65	0.48	8.84
Sz	583.27	87.78	81.75	23.20	96.98
Sv	476.17	35.96	36.68	20.41	49.67
Sp	107.11	51.83	45.07	2.79	47.31
Spd	13.28	33.26	49.08	0.40	159.43
Spc	6104.10	200.47	126.97	94.43	116.23

The values of the parameters Ra and Sa are approximately the same, which proves the accuracy of the measurements. These are averaged roughness height parameters, which do not respond to local irregularities (protrusions, depressions) on the surface. The highest values of these parameters were measured on the samples marked R (as-printed material surface). On the other contrary, the lowest values were determined on samples O – tumbled surfaces, where Ra = 0.44 µm and Sa = 0.48 µm. For samples P, K and M, the values ranged from 7.25-8.84 µm for both the profile roughness parameters Ra and the surface roughness parameters Sa.

Comparison of the roughness parameters Rz of the maximum height of the profile and Sz of the surface, shows the advantages of evaluating the surface topography using surface parameters, where the measured height maxima on the surface reached double values for samples P, K and M and up to five times values for samples R and M.

The lowest number of peaks per unit area was achieved on the surface of tumbled sample O, where the Spd value was 0.40 mm^{-2}. At the same time, the value of the parameter Spc = 94 mm^{-1}, indicating the largest radius of curvature of the elements on the surface. We are referring to the specimen with the highest surface quality achieved. On the contrary, the highest value of the parameter was determined for sample R, where Spc = 6104.10 mm^{-1}. Such a high value of the arithmetic mean of the peak curvature indicates a very pointed shape of the individual protrusions on the surface.

3.3 Thickness of the vitreous enamel coating

The thickness of the enamel coating was measured by a non-destructive method, according to the EN ISO 2808 standard. The measurements were carried out with an Elcometer 456 thickness gauge, which is based on electromagnetic induction. The thickness of the coating is

determined by the changes in the magnetic field after the probe is applied to the ferromagnetic substrate.

The probe used is capable of measuring both ferromagnetic and paramagnetic substrates. The probe of the instrument is positioned perpendicular to the coating to be measured and the thickness is determined from the change in magnetic flux.

Measurements were always taken in three areas on all samples, as indicated in Figure 2.

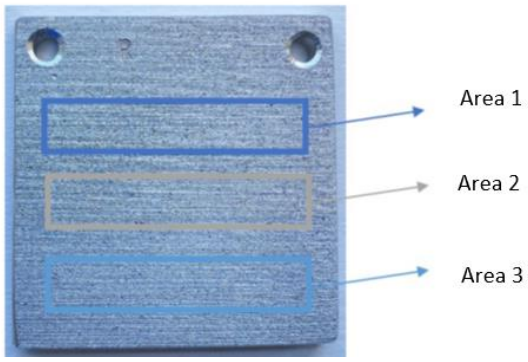


Fig. 2: Measured areas

Three measurements were taken in each area and then averaged. Since the application of the enamel slurry was done by dipping technique, the thicknesses in different areas may vary and there may also be wedging of the coating.

The manufacturer's recommended thickness of the enamel layer was stated to be approximately 100 µm. Higher coating thicknesses may result in surface defects, which have been experimentally verified (Figure 3). If the coating thickness values were well below 100 µm, it is possible that the coating would not perform well in terms of durability.

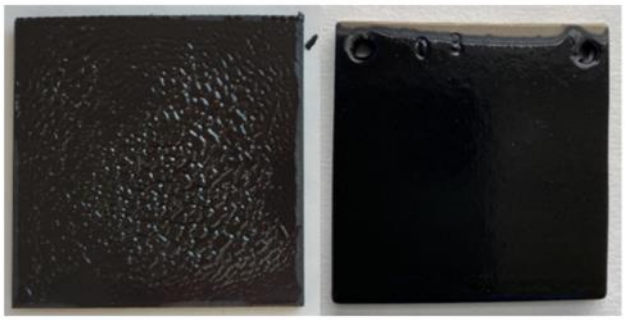


Fig. 3: Sample with defects (left), sample without defects (right)

Figure 3 on the left, shows a sample blasted with Polgrit, where a different technology – spray coating – was used to apply the enamel coating. The measured thickness values were above 200 µm. It is the double thickness of the coating compared to the manufacturer's recommendation that leads to orange peel type defects and to visibly damaged surface. In contrast, Figure 3, right, shows the specimen after mechanical pre-treatment – tumbling and dip coating technology that was used in this article. The thickness values on this sample were approximately 100 µm. The following table (Table 2) contains the measured values of coating thickness for all samples (R, P, K, O, M).

Tab. 2: Coating thickness – measured mean values

Sample	Thickness [µm]	
	Measured Mean Value	Std. Dev.
R	118.44	13.97
P	101.22	10.35
K	123.44	9.49
O	67.11	8.61
M	111.66	6.58

The measured coating thickness values for all samples (R, P, K, O, M) (Table 2) are then processed graphically for visual comparison in a box plot, see Figure 4.

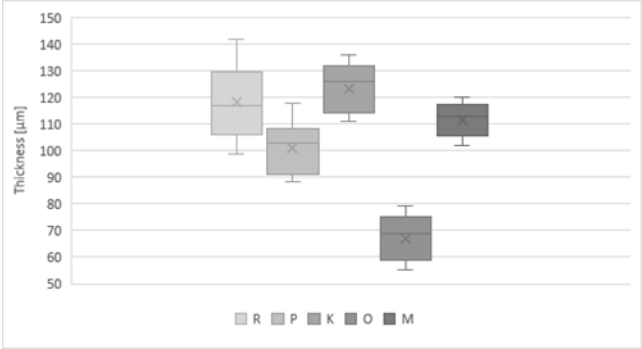


Fig. 4: Box plot of measured coating thicknes

Figure 4 shows box plot of the measured coating thicknesses on all samples. For the R as-printed samples we can observe the widest dispersion of the measured thickness values. For the O samples, plastered in the plastering machine, it can be seen that the coating thickness values were around 70 µm. The low thickness was due to the low anchoring profile of the smooth surface. In terms of the thickness requirements recommended by the enamel powder manufacturer, the P samples, blasted with Polgrit abrasive, performed the best.

4 CONCLUSIONS

Based on the experimental tests performed on the pretreated samples, the optimum surface pretreatment was recommended. The R samples have excessively high roughness, which could have a negative effect on the non-uniformity and the resulting quality of the enamel coating. The tumbled samples had a surface roughness that was too low. The enamel slurry was more difficult to apply to these pre-prepared samples than to the other samples. The thickness of the coatings on these samples also was the lowest. The ideal surface roughness was found in samples P, K and M. In the pickled samples (M), the surface was etched with hydrochloric acid to form unevenness and pores. Of all the pretreatments performed, blasting performed the best, based on the performed measurements. Polgrit blasting was evaluated as the best surface pretreatment prior to enamel coating application due to the creation of a defect-free coating of defined thickness, aided by the anchoring profile created by this pretreatment method.

5 ACKNOWLEDGMENTS

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