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MECHANISMS OF STRENGTH PROPERTIES ANOMALY OF Fe-Al SINTERS BY COMPRESSION TESTS AT ELEVATED TEMPERATURE

O MECHANIZMACH ANOMALNEGO WZROSTU WŁAŚCIWOŚCI WYTRZYMAŁOŚCIOWYCH PRZY ŚCISKANIU SPIEKÓW Fe-Al W PODWYŻSZONEJ TEMPERATURZE

The results of uniaxial compression test of Fe-Al sinters at a strain rate of 2×10^{-3} s^{-1} was presented in this paper. Compression tests were performed at room temperature and at elevated temperature in the range of 200 to 800°C in air. Sinters were obtained by liquid phase sintering resulted in production of FeAl intermetallic matrix with uniformly spaced $Al._2O_3$ dispersoid located at the grain boundaries of the intermetallic matrix.

Typical stress-strain curves were obtained from compression tests. Thus, the yield strength, compression strength and ductility were determined. The anomalous compression strength peak was observed at temperatures in the range of 300–700°C which was accompanied by a gradual (over 500°C) increase in ductility and gradual (over 400°C) decrease in compressive yield strength.

An assumption was made, that the reason for this anomaly was competitive interaction between thermaly activated phenomena, recovery material, and work hardening (dominating up to 700°C) of sinters during deformation.

Comparison of the yield strength data with deformation data at different temperatures showed that sinters have optimal values of these parameters at temperatures in the range of 400–600°C.

Keywords: Fe-Al intermetallic sinters, hot deformation, microstructure, mechanical properties

W pracy przedstawiono wyniki badań spieków Fe-Al poddanych statycznej próbie ściskania w warunkach jednoosiowego obciążenia z szybkością odkształcenia $2\cdot10^{-3}$ s^{-1}, w temperaturze pokojowej oraz w temperaturze z przedziału 200 do 800°C, w atmosferze powietrza. Badane próbki otrzymano metodą spiekania z udziałem fazy ciekłej, co pozwoliło na uzyskanie struktury składającej się z intermetalicznej osnowy FeAl i drobnych tlenków Al_2O_3 rozmieszczonych równomiernie po granicach ziaren osnowy.

Na podstawie otrzymanych krzywych ściskania wyznaczono wartości: $R_{c0.2}$, R_c i A_c, obserwując w zakresie temperatur 300–700°C anomalny wzrost doraźnej wytrzymałości na ściskanie, któremu towarzyszy stopniowy (powyżej 500°C – intensywny) wzrost plastyczności i stopniowe (od 400°C) obniżanie granicy plastyczności. Wysunięto hipotezę, że przyczyną obserwowanego zachowania jest konkurencyjne oddziaływanie zjawisk aktywowanych cieplnie uplastyczniających materiał i umocnienia odkształceniowego (dominującego aż do 700°C) materiału spieków podczas ściskania. Porównanie zmiany wytrzymałości doraźnej na ściskanie R_c i umownej granicy plastyczności $R_{c0.2}$ w zależności od temperatury odkształcenia wykazało, iż szczególnie korzystny przedział temperaturowy potencjalnego zastosowania badanych spieków występuje w zakresie 400–600°C, zapewniając maksymalny poziom wartości R_c ok. 1100 MPa, przy korzystnym poziomie $R_{c0.2}$ wynoszącym powyżej 500 MPa i skróceniu względnym do chwili wystąpienia pierwszych pęknięć wynoszącym 7–12%.

1. Introduction

FeAl intermetallic phase-based sintered materials constitute a promising group of modern engineering materials due to their attractive physical and mechanical properties as well as low cost of raw elements [1]. The production technology based on powder metallurgy eliminates a number of limitations in the manufacturing process [2], allowing for complicated shapes of the semi-finished or finished products. Furthermore, the production technology enables a wide selection of various chemical compositions, eliminates a problem of the grain coarsening, inhomogeneity of chemical composition and structure and finally, secures for easier control of the structure of material, as well as reduces the use of raw elements [3–5].

It has been found that temperature and pressure are the primary parameters influencing the production of

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FeAl sinters from elemental aluminum and iron powders. To reduce the cost and shorten the duration of the process while retaining advantageous morphological structure which results, in turn, in good combination of the used parameters of sinters, the following process route, consisting of three main stages, is suggested:

– uniaxial cold pressing of the powder mix under high pressure;
– initial sintering with the presence of liquid phase under pressure;
– homogenization.

Sinters obtained by the suggested process were deformed in compression tests at room temperature and at temperatures in the range of 200 to 800°C in air. The mechanical properties of the sinters were determined. Additionally an optimum working temperature has been found for potential high-temperature application of these sinters.

2. Experimental procedure

Elemental powders of 99.8% pure iron with the average particle size of 200 μm and 99.6% pure aluminum with the average particle size of 70 μm, pre-mixed in a stoichiometric ratio of 50at.%Fe-50at.%Al were mixed together by ball milling. Subsequently, the powder blend was consolidated by uniaxial cold pressing (Fig. 1a) and sintered under constant pressure of 50 MPa at temperatures above 900°C with the presence of liquid Al phase (during presintering) and the presence of different phases such as: Fe, Fe_3Al, Fe_2Al_5, $FeAl_3$, FeAl phases after cooling of presintered material (Fig. 1b). Final microstructure of iron aluminides based on ordered (B2) FeAl intermetallic phase after homogenization stage was shown in Fig. 1c.

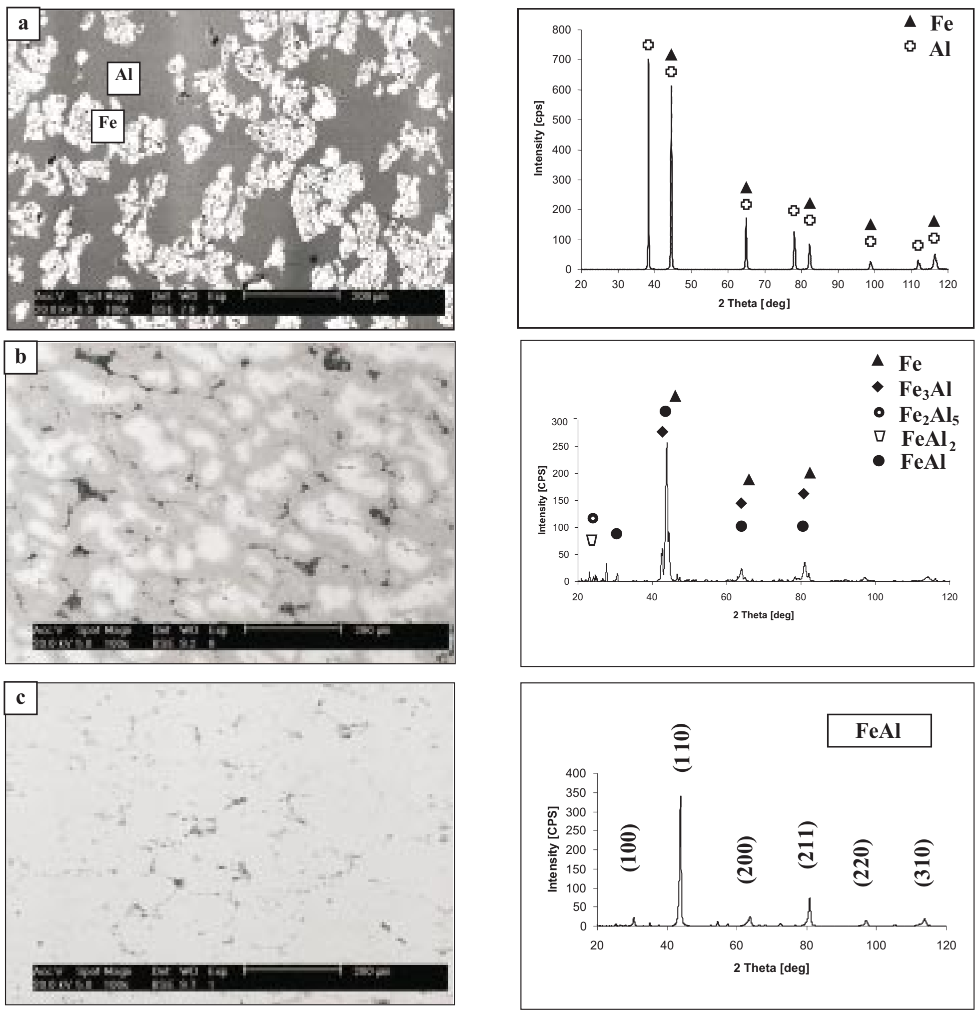

Fig. 1. Morphology and X-ray analysis of Fe and Al powders mixture after uniaxial cold pressing (a), Microstructure and phase analysis of material after presintering process at chamber temperature of 900°C/5h (b), Microstructure and phase analysis of material preliminary sintered at temperature of 900°C/5h and homogenized at 1100°C (c)

The analysis of structure and chemical composition in sinter micro areas was carried out with PHILIPS XL30 (LaB6) scanning microscope equipped with the X-ray DX4i/EDAX microanalysis device. XRD phase analysis was performed on a 3003TT Seifert diffractometer with the access to using PDF-2 database for diffraction pattern identification. Cu Kα_1 radiation (λ = 0.15405 nm) was used in this study produced at an accelerating voltage of 40 kV and a current of 40 mA. The scan range angle was from 2θ = 20 deg to 120 deg and the scan rate was 3s at the step size of 0.02 deg. The compression test was carried out with controlled strain rate of 2×10^{-3} s^{-1} on Instron 8802 tester equipped with Instron 2620-601 dynamic high-temperature extensometer (Fig. 2.). True strain of samples were also measured by this extensometer. The compression test was carried out in pipe furnace with temperature control. Samples were covered with graphite grease in order to minimize shearing stress and were introducted to furnace chamber after heating it to suitable temperature. This test was conducted after 20 minutes of keeping the samples in constant temperature. Immediately after the test the samples were air cooled.

Fig. 2. Sintered sample between Instron push – rods equiped with high – temperature extensometr

3. Results and discussion

Typical true stress-true plastic strain curves recorded at different temperatures are shown in Fig. 3. For each specimen, the yield strength, compression strength and ductility (unit shortening) was computed. These values are listed in Table.

Fig. 4 shows that the value for yield strength at temperatures in the range RT to 300°C is relatively high and comparable with value of compression strength. Simultaneously, low value for ductility sugested about brittle failure of sinters (Fig. 5). In the range 300-400°C, $R_{c0,2}$ value reduces rapidly with minimal increase of ductility. The same behaviour of yield strength value was observed by R. Carleton ET AL. [6] in similar temperatures range. They examined by tensile test of cast and hydroextruded samples (at 800°C) of Fe – 48% at. Al – 0.12% B at alloy. Nevertheless the autors [6] did not explain reasons of this FeAl behaviour.

TABLE

Influence of compression tests temperatures on strength parameters of FeAl 50 intermetallic sinter obtained by pressure – assisted presintering at 900°C and pressure – less homogenization at 1100°C, 5h

Compression temperature [°C]	Compression yield strength $R_{c0.2}$	Compression strength R_c [MPa]	Unit shortening [%]
RT – a	820 ± 86	875 ± 12	3.1 ± 2.1
200 – b	821 ± 45	882 ± 33	3.5 ± 1.1
300 – c	791 ± 37	932 ± 27	4.4 ± 0.7
400 – d	567 ± 61	1022 ± 137	5.9 ± 2.4
500 – e	540 ± 58	1093 ± 129	7.2 ± 3.2
600 – f	496 ± 97	1112 ± 139	12.8 ± 6.0
700 – g	392 ± 73	1072 ± 88	26.9 ± 15.5
800 – h	274 ± 99	694 ± 156	36.1 ± 1.3

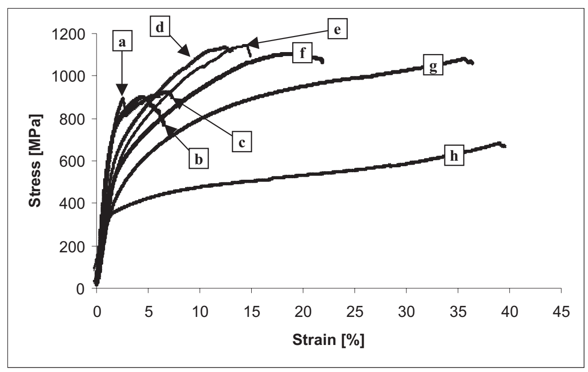

Fig. 3. Typical true stress – true strain plots for Fe-Al 50% at. sinters compressed at different temperatures (description in Tab. 1)

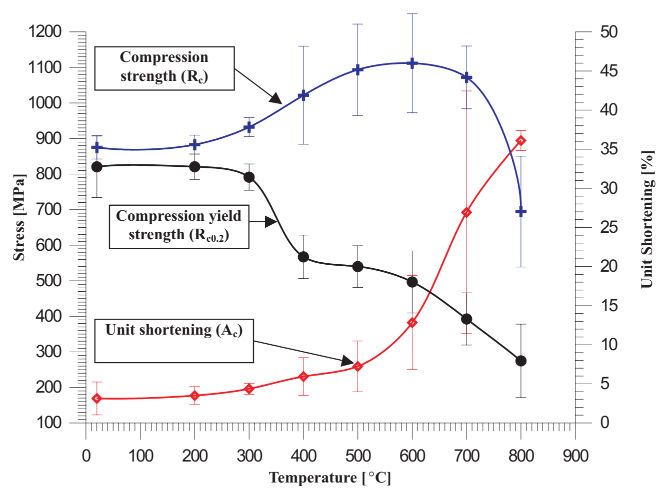

Fig. 4. Compression strength parameters of FeAl 50 sinters versus compression test temperature

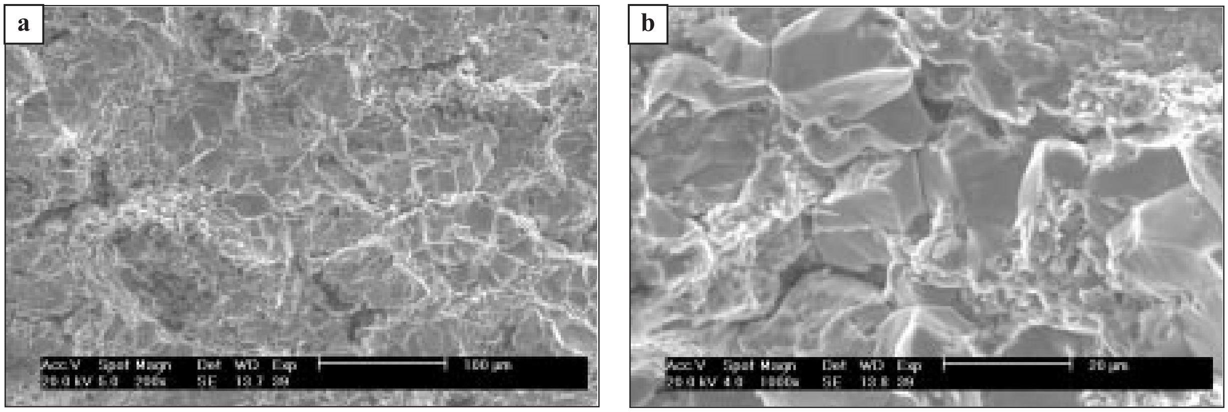

Fig. 5. Typical fracture morphology of FeAl 50 sinter after compression test at room temperature, magnification 200x (a), 1000x (b)

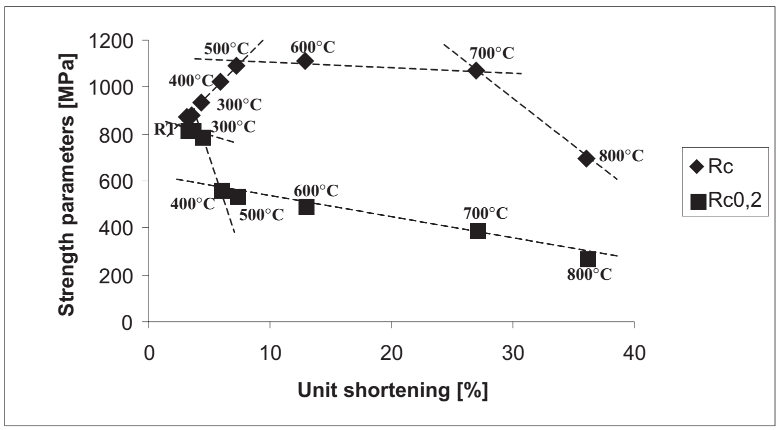

Fig. 6. Plots of compression strength (R_c) and compression yield strength ($R_{c0.2}$) versus unit shortening for compression at different temperature

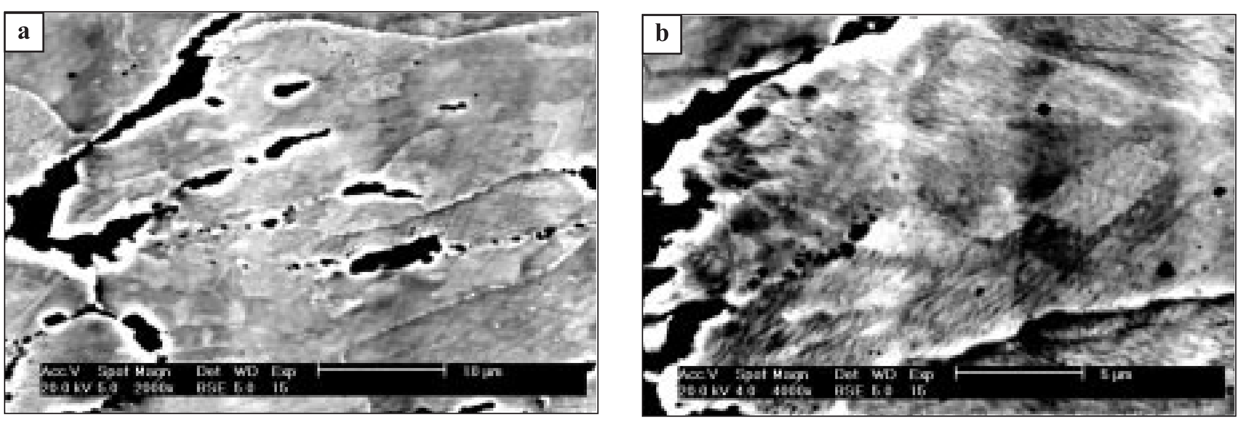

Fig. 7. Structure of FeAl 50 sinter after compression test at temperature of 300°C (visible cavities of etching confirm a presence of dynamic recovery during compression)

Dynamic recovery proceeding simultaneity with deformation is probably the cause of the observed effect. This results in characteristic decrease of excess point defects and anihilation of dislocation structure by cross slip or climbing of dislocation. Fig. 7 shows cavities of etching in microstucture of sinters after deformation at temperature of 300°C, which are residue of poligonization structure.

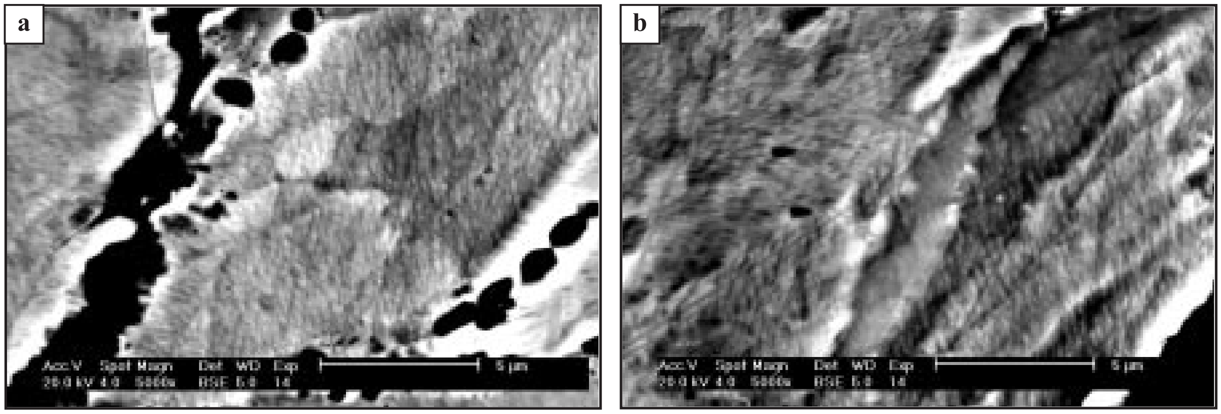

Fig. 8. Structure of FeAl 50 sinter after compression test at temperature of 800°C (presence of small equiaxed grains confirms dynamic recrystalization process during compression)

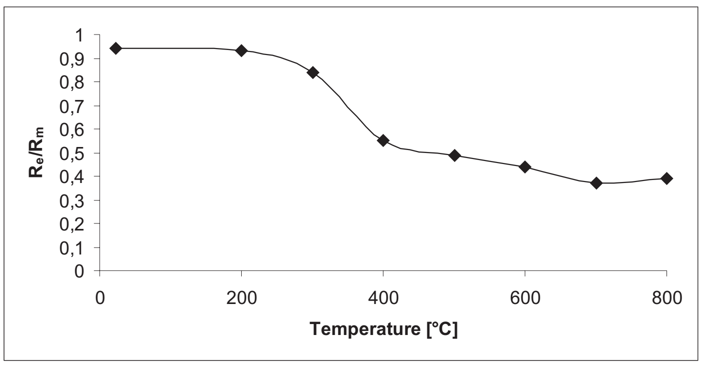

Fig. 9. Coefficient of elasticity values versus temperature of compression test

According to W y r z y k o w s k i et. al. [7] plastic deformation at elevated temperature ($T>0.25T_m$) causes that an increase in dislocations is higher then the number of dislocations annihilated during recovery. In connection with them value of compressive strength increases together with increase of ductility and decrease of yield strength (up to 600°C). It results in strain hardening of sinters structure.

Heterogeneity of long range ordering parameters (LRO) except of hardening connected with rebuilding of dislocation structure, results in decrease of compressive strength and yield strength values when temperature of compression test increases to 600°C. R.W. C a h n, in his article from 1998 [8], turned attention that small changes LRO could have influence on mechanical properties of intermetallics. Thermal fluctuations of LRO and differences of the speed of ordering process resulting from inmonotonic changes of thermal vacancies concentration or incomplete incident order ⇔ disorder transition can be a cause of such inhomogeneity. The change of LRO causes change of energy of antiphases boundary uniting partial dislocations in a single superdislocation. Of above mentioned growth of antiphases boundary energy caused by enlargement of LRO parameter of FeAl intermetallic sinter structure, could be additional cause of sinter hardening observed up to 600°C.

However, in available literature, there are no data proving direct influence of LRO parameter on mechanical proprieties of FeAl alloys. That is why also, the full interpretation of showed effects needs farther, detailed investigations in this area.

In the range of 500-700°C temperature (Fig. 4, 6) value of compression strength is about constant – hardening is balanced by softening of material connected with recovery and (over 700°C) recrystalization, causing reduction of dislocation density.

Decrease of compression strength value above 700°C indicates that thermal activated processes start to play a more significant role than strain hardening. Fig. 8 shows the new grains created by dynamic recrystallization. Simultaneously, about 700°C the <111>{110} to <100>{110} transition of slip system transition takes place [9]. Fig. 4. shows increase of value of ductility.

Increase of temperature at which compression is conducted causes simultaneously decrease of coefficient of elasticity value defined as R_e/R_m ratio from 0.94 (RT) to 0.39 (800°C) – Fig. 9. Nevertheless the biggest influence of compression temperature on coefficient of elasticity is observed between 300-400°C where dynamic recovery does not work hardening during plastic deformation of these materials.

4. Summary and conclusion

Study of FeAl sinters microstructure and results of compression tests can lead to the following conclusions:

1. FeAl – based intermetallic sinters obtained from elemental Fe and Al powders mixture by cold pressing, presintering with presence of Al liquid phase and homogenization showed anomalous strength peak during compression at temperature range of 300–700°C.
2. Competitive interaction between thermal activated phenomena and strain hardening was recognized as hypothetical reason of intense increase of compression strength (up to 700°C) acompanied with gradual (over 500°C) increase in ductility and gradual (over 400°C) decrease in compressive yield strength.
3. It was affirmed, that particularly profitable temperature range of the potential use of investigated sinters includes 500–600°C assuring maximum level of compression strength value (near 1100 MPa) advantageous level of yield strength (above 500 MPa) and satisfactory level of plasticity (7–12% at compression).
4. More reliable SEM and TEM examinations are necessary to characterize phenomenon of anomaly increase of mechanical properties of FeAl – based sinters obtained by suggested production technology, at elevated temperature.

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