Jernkontorets forskning

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METALLISKA MATERIAL

Guidelines:
Corrosion Assessment of Additively Manufactured Stainless Steels

METALLISKA MATERIAL

GJUTERIFÖRENINGEN Swedish Foundry Association

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Med stöd från

Guidelines:

Corrosion Assessment of Additively Manufactured Stainless Steels

Participants:
Sandvik Additive Manufacturing, Quintus Technologies AB,
Alfa Laval Technologies AB, Swerim AB

Summary

The aim of these guidelines is to provide an introduction for working with corrosion evaluation of additively manufactured stainless steel (AM SS). Baselines for four standard test methods, ASTM G150 / ISO 17864, ASTM G61 / ISO15158, ASTM G48 and ISO 18069 are presented, together with recommendations for specific modifications to assess AM SS. These guidelines are aimed particularly towards companies who are new in the field of AM SS.

The guidelines were developed within the AMCO project (Additively manufactured stainless steel products in corrosive environments, 2018-03910, Vinnova SIP Metallic Materials) in which tools for validation of corrosion properties of AM materials were developed and the applicability of existing standards for corrosion testing to additively manufactured parts was assessed. Two common stainless steel materials were investigated: the austenitic 316L and the super duplex 2507, produced with AM Powder Bed Fusion – Laser Beam (PBF-LB).

As-built, heat treated and abrasively **blasted** | As-built, heat treated and **pickled** | As-built, heat treated and **tumbled**

Macroscopic images of as-built heat-treated PBF-LB/SDSS 2507 surfaces after different surface treatments: blasting, pickling, and tumbling.

MAIN OBSERVATIONS FOR CORROSION IN AM SS

- Post-processing (heat and surface treatments) is necessary for AM SS material performance.
- Post-processing parameters (e.g. cooling rate) are critical for material performance.
- As-built printed surfaces and conventional SS surfaces are not directly comparable.
- Surface oxides formed during printing or post-processing must be removed to achieve a final product with sufficient corrosion resistance.
- Corrosion properties are strongly influenced by surface condition (as-built, ground, blast, pickled, tumbled etc.).
- Pickling procedures for conventional SS may need to be modified for AM SS.

GENERAL FACTS ON CORROSION

Corrosion facts related to costs[1]

- Global cost of corrosion: Ca. $ 2.5 trillion, c. 3.4% of a country's GDP
- Annual cost of corrosion in Sweden: Ca. $ 17 billion
- Annual cost per person: Ca. $ 1 670

Corrosion facts related to global environmental impact[2,3]

Corrosion can jeopardize human safety, cause plant shutdown, waste time and resources, reduce efficiency, involve expensive maintenance, cause overdesign, inhibit technological progress and cause loss or contamination of product[2]. For example, corrosion can cause leaking tanks, containers and pipelines which can result in contamination of the environment[3].

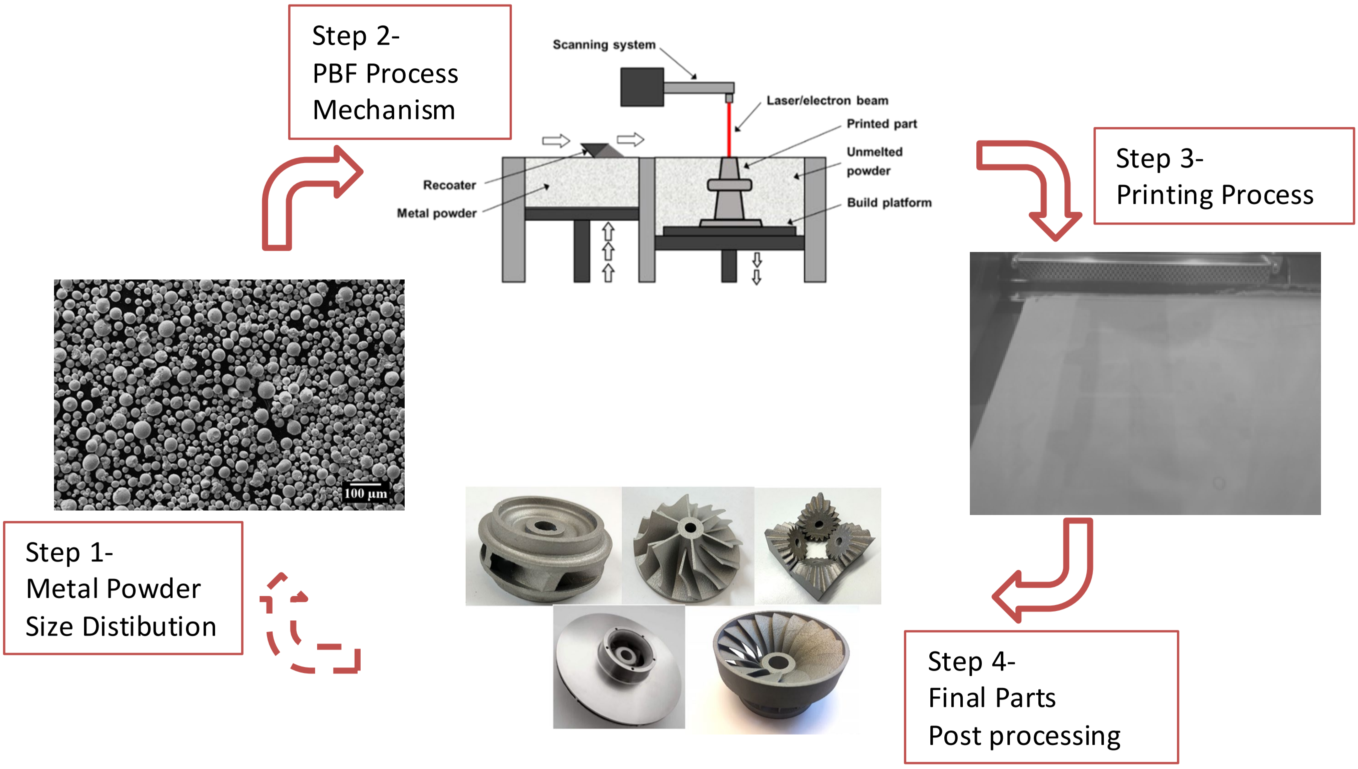
Stainless steels

Stainless steels come in numerous different types, more specialized SS such as duplex SS are more sensitive to heat treatments than standard SS, such as 304 or 316.
Stainless steels have multiple benefits such as:

- Less need to replace corroding components
- Less need for maintenance
- Recyclable
- Hygienic

Why AM SS?

- Resource-saving
- Cost efficient
- Ability to build specific and complex designs on demand
- Tailored microstructures



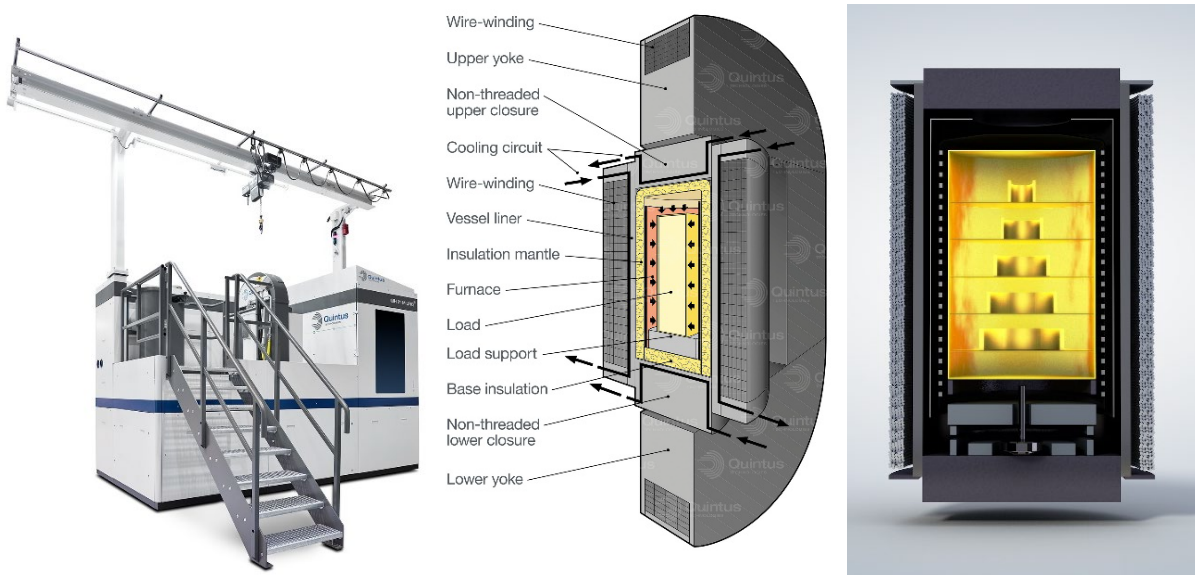
Schematic image of the PBF-LB AM-process, Courtesy of Sandvik

Additive manufacturing (AM)

Additive manufacturing (AM) processes are relatively new and offer the possibility to manufacture complex design, mass customization and manufacturing on demand and thereby reduce the working capital. Among various AM technologies, the powder bed fusion process depicted above is one of the most common. The process of manufacturing components using the Powder Bed Fusion – Laser Beam (PBF-LB) process starts with the selection of the metal powder. These metal powders usually have a particle size distribution between ca. 15µm and 50µm and are atomized using a specific powder production technique. In the next step, (1) a powder layer is spread uniformly on the build platform using a roller or recoater and then (2) sliced CAD data of the part is locally traced and melted by the laser. The process is repeated (3), and the part is produced layer by layer- and hence the term as additive manufacturing. After printing the part inside the chamber is moved to post processing operations (4). Furthermore, the non-melted metal powder is removed and sieved to the optimum particle size distribution and can be reused for the next build job with suitable quality control methods.

It must be noted, that AM processes very often use different process parameters for contour and bulk regions of the manufactured parts result in different microstructures. To assess the bulk corrosion properties, the contour areas need to be removed thoroughly.

In this study the PBF-LB method was used to produce parts and specimens from Osprey® 2507 (Super Duplex Stainless steel) metal powder using particle size distribution of ca. 15-50µm.



Left: HIP machine QIH21. Center: Schematic of HIP-equipment. Right: Cross-section of the HIP furnace. Courtesy of Quintus Technologies AB.

Hot Isostatic Pressing (HIP)

HIP has been used for many years to close porosity that is not connected to the surface and to heal defects in powder materials by using high pressure in combination with elevated temperature for a given time. Production of metallic components using additive manufacturing (AM) is developing quickly, and HIP is often used to heal porosity and other defects to improve density and subsequently mechanical properties such as ductility and fatigue resistance. More recent equipment developments have led to introduction of heat treatment capability whilst still under pressure in the hot isostatic press (High Pressure Heat Treatment, HPHT™). High speed cooling in conjunction with increased steerability of the process have led to the development of tailored HIP cycles to preserve the fine grain size seen in AM processes and/or to obtain the desired phase fraction, thereby maintaining strength whilst improving fatigue life.

In this study, specially tailored HPHT™ cycles were applied to HIP and heat treat two different stainless steel alloys to optimize mechanical and corrosion properties.

Selected Standards for Corrosion Testing

ASTM G150[4] / ISO 17864[5]: Electrochemical critical pitting temperature testing

- Environment: 1M NaCl solution
- Procedure: Apply a potential of 700 mV vs. SCE using a potentiostat
- Result: **C**ritical **P**itting **T**emperature **CPT**

ASTM G61[6] / ISO 15158[7]: Cyclic potentiodynamic polarization measurements for localized corrosion susceptibility

- Environment: 1M NaCl solution
- Procedure: Performing polarisation sweeps using a potentiostat while measuring resulting currents
- Result: **C**ritical **P**itting **P**otential **CPP**

ASTM G48 Method E[8]: Critical pitting temperature test

- Environment: 6 wt.% $FeCl_3$ with 1 wt.% HCl at defined starting temp.
- Procedure: Immersion tests at increasing temperatures
- Result: **C**ritical **P**itting **T**emperature **CPT**

ISO 18069[9]: Determination of uniform corrosion rate in liquids

- Environment: Intended solution at a desired temperature
- Procedure: Immersion tests for a defined time
- Result: Corrosion rate [mm/a]

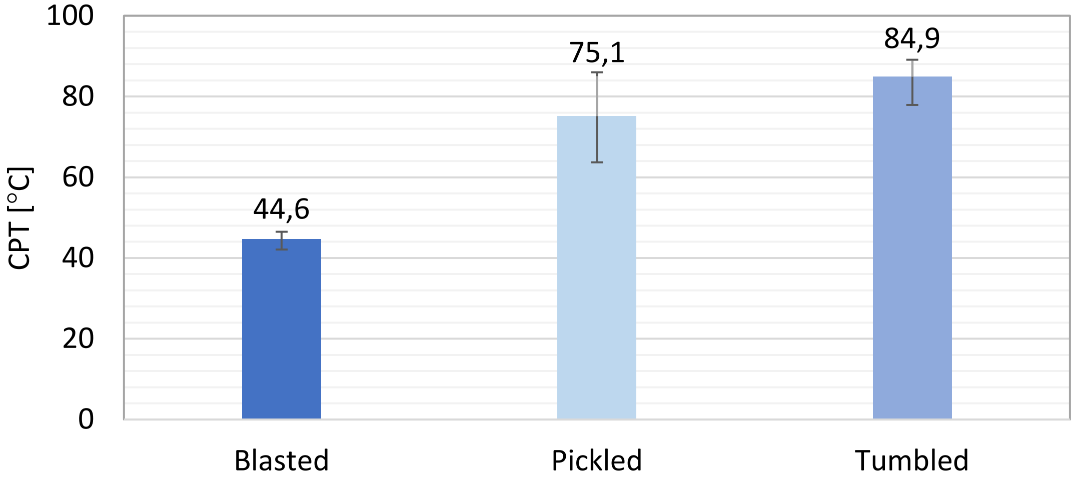
Aspects to Consider

- The test surface shall be free from heat tints, loose particles, oil, grease etc.
- Ultrasonic cleaning in an inert liquid should be used before testing to remove loose particles (metal powder, blasting media, etc.).
- Surface state:
  - Presence of oxides
  - Surface roughness (if not prepared according to the standard)
  - Regard differences in surface finish and state when comparing different samples.
- It is necessary to probe pit sites on the metal surface with a needle to expose corrosion attack under the surface. Microscopy must be used to confirm pitting.
- CPT can be used for ranking purposes and quality quantification but should not be a basis for material selection.

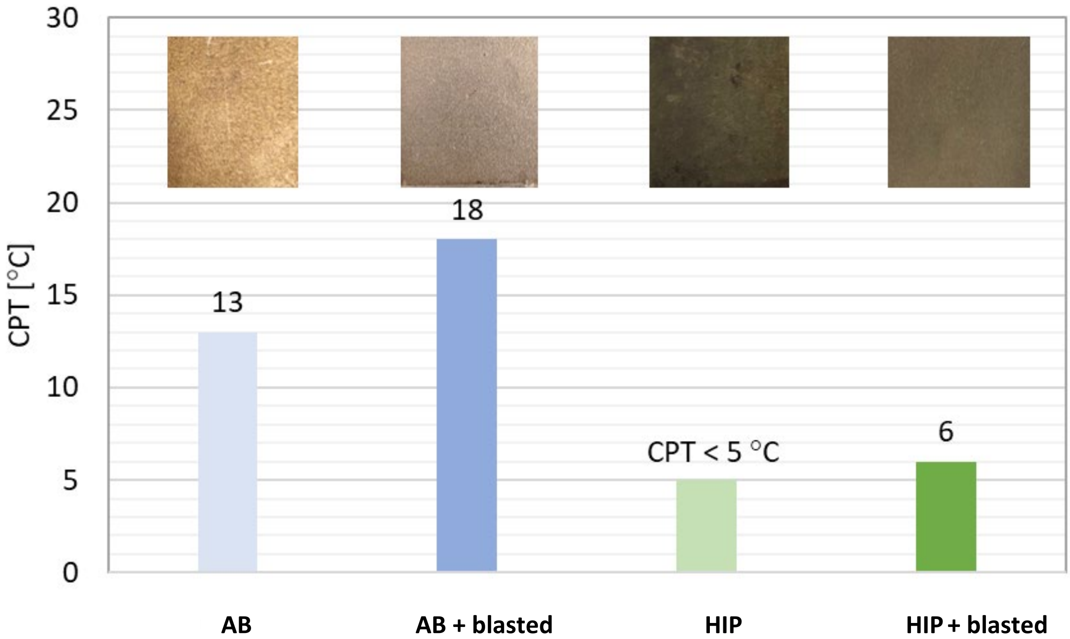
For ASTM G150 / ASTM G61

- Sample geometry: flushed-port cell (standard) or, for complex geometries: immersed samples (not standard)
- A surface that is not prepared according to standard may cause an initial current increase followed by repassivation.

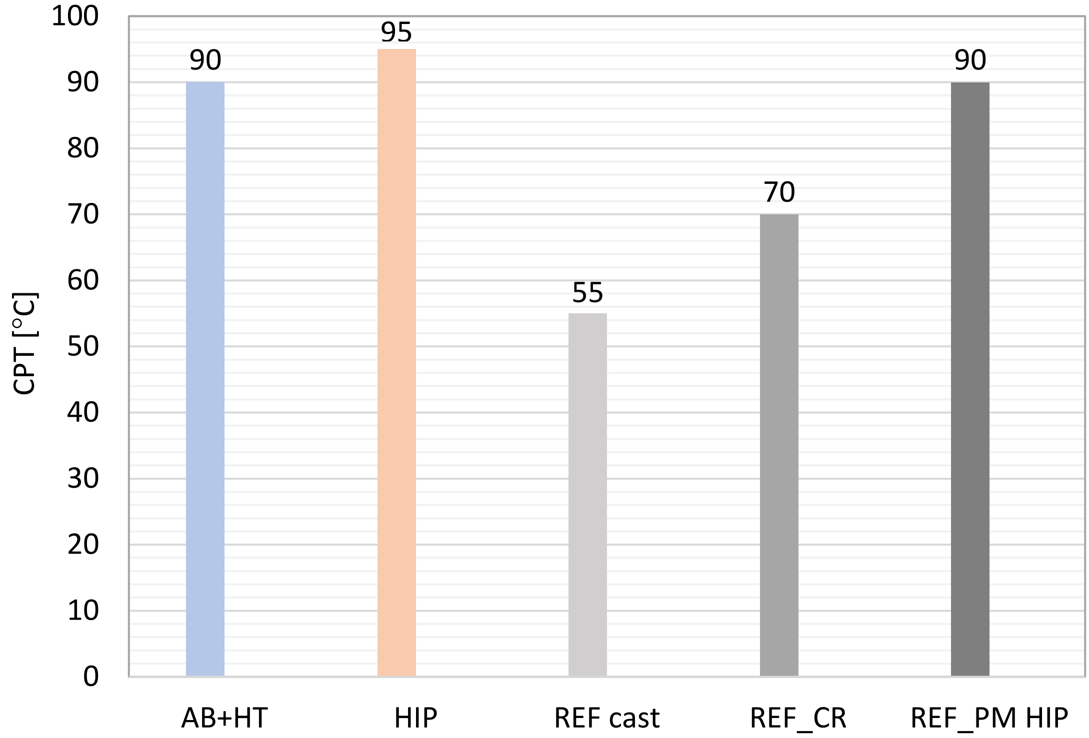
EXAMPLES



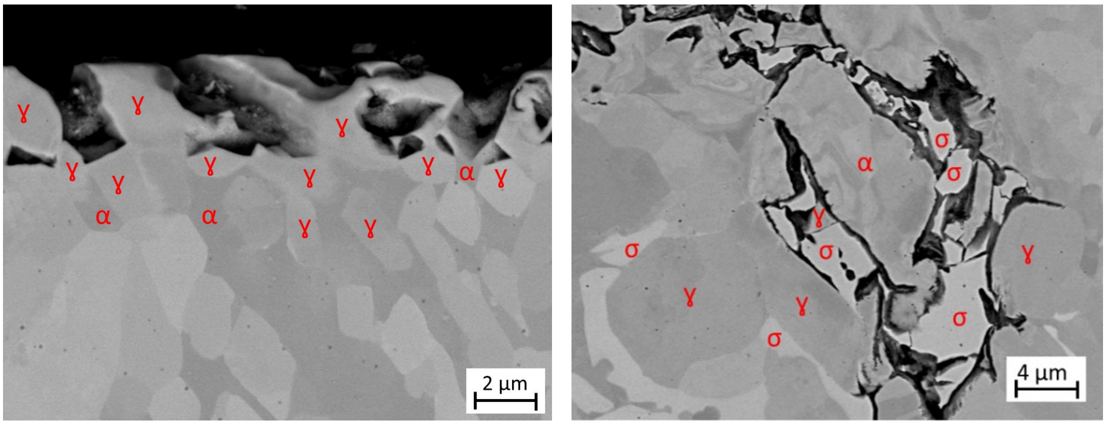
*CPT values for as-built and heat-treated (AB+HT) PBF-LB/SDSS 2507 material (**surface evaluation**) after (1) blasting, (2) pickling and (3) tumbling. Testing in accordance with **modified ASTM G150**[4] in 1M NaCl. Super-duplex stainless steel 2507, UNS 32750, specimens were prepared from Sandvik's Osprey® 2507 VIGA powder at Sandvik Additive Manufacturing.*



*CPT values after HIP **with significant high temperature surface oxides**, and as-built (AB) PBF-LB 316L samples, with and without blasting including pictures of the corresponding surfaces. Testing in accordance with **modified ASTM G150** in 1M NaCl on nonstandard surfaces. Stainless steel 316L, UNS S31603, test specimens, were printed at GE Healthcare. HIP was conducted at Quintus Technologies.*



*CPT values for **horizontal cross-sections** as-built and heat-treated (AB+HT) and HIP PBF-LB/SDSS 2507 material compared with reference cast, cold rolled (CR), and PM HIP SDSS. Testing in accordance with standard **ASTM G48 method E**[8]. Super-duplex stainless steel 2507, UNS 32750, specimens were prepared from Sandvik's Osprey® 2507 VIGA powder at Sandvik Additive Manufacturing. HIP was conducted at Quintus Technologies.*



*SEM-BSE images of **cross-sections** of PBF-LB/SDSS 2507 showing corrosive attack in the ferritic phase (α) in the as-built and heat treated (AB+HT) material (left) and around the sigma phase (σ) in a material that underwent an unsuitable heat treatment (right). After immersion testing in sulfuric acid according to **ISO 18069**[9]. Super-duplex stainless steel 2507, UNS 32750, specimens were prepared from Sandvik's Osprey® 2507 VIGA powder at Sandvik Additive Manufacturing.*

Adaptions for AM SS

ASTM G150[4] and ASTM G61[6]

- Complex geometry → Immersed samples
  - The standard test is performed on a ground surface and is suitable for analysis of bulk material. Coarser surfaces, non-flat samples or samples that are too big to fit the cell are not suitable for the flush port cell setup and might cause leakages. A modification where the sample is masked to expose a specific area and then suspended in the test solution can be employed to obtain ranking of samples with a rough surface or different geometry. However, this must be done in a way so that no crevice corrosion is formed.
- Testing any surface that is not prepared according to standard, 600-grit, is a type of modification of the test. Always state the surface finish of the tested material (e.g. as-built, postprocessed, ground, polished, blasted, pickled).
- Highly corrosion resistant alloys
  → change to more aggressive environments (3M $MgCl_2$), and/ or increase the temperature.

ASTM G48 Method E[8]

- Testing of specimen polished according to the standard for bulk material is relatively straightforward. However, since the AM material may contain pores, all specimen surfaces should be documented before the corrosion test to avoid mistaking pores for corrosion pits.
- Testing of as-built surfaces or surfaces treated with processes like abrasive blasting, tumbling, or pickling requires special consideration.
  - Abrasive blasting and other surface treatment processes can result in contamination of the AM surface. The contamination can have a negative impact on the actual corrosion resistance and on the corrosion test. Control the surface cleanliness of the produced articles and test specimen with suitable method (e.g. SEM-EDS).
  - Examination of pitting on those coarser surfaces is not as straightforward as on polished surfaces. For the evaluation it is recommended always to have parallel unexposed samples for comparation.
  - The tested surface finish should always be stated in the report.
- The given recommendations are not only useful for Method E but may be applicable for other methods in ASTM G48.

ISO 18069[9]

- Highly corrosion resistant alloys
  → Change to more aggressive environments, and/ or increase the temperature.
- Always state the surface finish of the tested material (e.g. as-built, postprocessed, ground, polished, blasted, pickled).
- Choose relevant reference material.

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