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Ancient and Modern Laminated Composites — From the Great Pyramid of Gizeh to Y2K

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Ancient and Modern Laminated Composites —

From the Great Pyramid of Gizeh to Y2K

(Abbreviated title: Ancient and Modern Laminated Composites)

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Abstract

Laminated metal composites have been cited in antiquity; for example, a steel laminate that may date as far back as 2750 B.C., was found in the Great Pyramid in Gizeh in 1837. A laminated shield containing bronze, tin, and gold layers, is described in detail by Homer. Well-known examples of steel laminates, such as an Adze blade, dating to 400 B.C.can be found in the literature. The Japanese sword is a laminated composite at several different levels and Merovingian blades were composed of laminated steels. Other examples are also available, including composites from China, Thailand, Indonesia, Germany, Britain, Belgium, France, and Persia. The concept of lamination to provide improved properties has also found expression in modern materials. Of particular interest is the development of laminates including high carbon and low carbon layers. These materials have unusual properties that are of engineering interest; they are similar to ancient welded Damascus steels. The manufacture of collectable knives, labeled "welded Damascus", has also been a focus of contemporary knifemakers. Additionally, in the Former Soviet Union, laminated composite designs have been used in engineering applications. Each of the above areas will be briefly reviewed, and some of the metallurgical principles will be described that underlie improvement in properties by lamination. Where appropriate, links are made between these property improvements and those that may have been present in ancient artifacts.

Introduction

In a recent review[1], the authors and their colleagues presented examples of historical laminated composites and described in detail modern studies of the mechanical behavior of laminated metal composites (LMCs). Laminated metal composites consist of alternating metal or metal-containing layers that are bonded with "sharp" interfaces. These materials represent a unique laminated or composite form that is different from graded materials, which have diffuse interfaces, or layered materials, in general, which can consist of alternating layers of a wide range of materials. Laminated metal composites can dramatically improve many properties including fracture toughness, fatigue behavior, impact behavior, wear, corrosion, and damping capacity; or provide enhanced formability or ductility for otherwise brittle materials. In many cases, through the choice of component materials, laminate architecture (such as volume percent of the component materials and layer thickness), and processing history, LMCs can be engineered to produce a material with prescribed properties.

In the present paper, the first section deals with the history of laminated composites. In particular, examples of their existence, composition, and structure are detailed. Extra emphasis is given to the Japanese sword both because of its complexity as a laminate at several levels, and its relatively well-documented history and the technical details that accompany that history. Included at the end of the section are some comments on modern knives that duplicate, in part, the ancient weapons. The second section of the paper first describes modern engineering applications of LMCs. Scientific and engineering studies on laminated composites are then presented — in some cases at the very thin-layer level, and in others at layer thicknesses that were close to those found in ancient laminates. Processing methods, as well as strength, durability, toughness, and damping properties, are discussed. The mechanisms of improving toughness by lamination are described in detail.

Where possible, the mechanisms leading to improved properties of modern engineered laminated composites are linked back to ancient artifacts.

History

The idea of laminating similar or dissimilar metals or alloys to form a composite material has been known from antiquity. The motivations for laminating metals are varied. For example, in carburizing the earliest forms of wrought iron, only thin layers could be carburized and so lamination was a way to create bulk material. (This could be the motivation for the most ancient laminates.) Another reason is that the hard material, steel, was rare and it was expedient to sandwich it between more common materials. (This motive is found in medieval knives.) From a mechanical viewpoint, optimizing the combination of strength, toughness, and sharpness is the basis for lamination. (Examples include the Japanese sword, the Halberd, and modern laminates.) Finally, there is a strong motivation based on decorative appeal. (Many modern knives are made in laminated form for this reason, but it could have been a motive in ancient knives also.) Some selected examples of laminated materials follow.

Laminated Iron Plate found at the Great Pyramid of Gizeh: In 1837, an iron plate (26 cm x 86 cm x a maximum thickness of 0.4 cm and weighing 750 g) was discovered by an excavation team near an air passage (Southern side) in the Great Pyramid at Gizeh, Egypt. The location of the plate was within an undisturbed section high up on the pyramid. The plate was removed to the British Museum and was not examined for its structure until El Gayer and Jones used modern metallographic techniques on a small (1.7 g) sample from the plate and published their findings in 1989.[2] A comment by Craddock and Lang[3] was included in the same issue of the Journal.

The significance of the plate is twofold. First, if it can be shown to be contemporaneous with the building of the pyramid, then it is one of the oldest known plates of iron metal ever discovered and dates from the 4th Dynasty, circa 2750 B.C. Second, the metallographic study of El Gayer and Jones revealed that the plate consists of:

> *...numerous laminates of wrought iron and that these laminates have been inexpertly welded together by hammering. The various layers differ from each other in their grain sizes, carbon contents, the nature of their non-metallic inclusions, and in their thicknesses.*

It was further deduced from elongated non-metallic inclusions that the welding process had been carried out at modest temperatures (~800°C) allowing re-crystallization of the iron matrix grains. The absence of metallic copper globules, and only small traces of elemental copper suggested that the plate had not been produced as a by-product of copper smelting operations of iron-rich copper ores. Also, a chemical analysis reported in 1926 revealed only trace levels of nickel, thereby confirming the plate to be of terrestrial (but not natural) origin rather than to be meteoric.[2] (It is noted that the above view on lamination is not universally agreed upon. An alternate view is that the heterogeneous nature of the plate is a direct result of a heterogeneous starting piece.[4])

Summarizing, El Gayer and Jones concluded that the iron pieces comprising the laminate were:

> *...intentionally produced during small-scale (and, possibly, very primitive) operations primarily designed for the production of iron metal (rather than copper metal). Furthermore, the presence of abundant inclusions of unreduced (or incompletely reduced) fragments of iron oxides in the metal laminations shows that the 'smelting' operations had been inexpertly carried out at low temperatures (probably between 1000 and 1100°C) and that the iron had been produced by the 'direct reduction' method – in which no molten iron is normally produced.*

And, most importantly, they also concluded:

> *Furthermore, the metallurgical evidence supports the archaeological evidence which suggests that the plate was incorporated within the Pyramid at the time that structure was being built.*

Although accounts by the excavation teams emphasize the fact that the plate was found within the pyramid, and is therefore contemporaneous with the pyramid, this view has not been generally accepted by archeologists.

Subsequent to the paper by El Gayer and Jones, the only other investigation of the plate came in 1993 by Craddock and Lang.[5] They agreed with the El Gayer and Jones study that the structure was similar to banded, wrought iron consisting of areas of varying carbon content. However, the absence of slag stringers and the presence of very large numbers of other inclusions, containing unusually high levels of Ca and P, led Craddock and Lang to a quite different conclusion regarding the method of manufacture (and therefore the origin and likely age) of the plate. They believe the structure to be one derived from "cast iron smelted with charcoal, and then treated by the finery process to remove the carbon and produce a solid lump or bloom of wrought iron." They go on to cite work proposing that this technique was the usual method of making iron in the post-medieval Islamic world. The Gizeh plate remains unusual, even in this scenario, because of the very high level of inclusions that it contains. Craddock and Lang do not, on the basis of their 1993 analysis, believe the plate is contemporaneous with the pyramid, concluding that: "the plate of iron from the Great Pyramid is of no great antiquity." Nonetheless, these authors confirm that if its age were to be contemporaneous with the pyramid that it would be "the earliest substantial piece of iron known," a finding accepted by the famous scientist Petrie in 1883.

Given these controversial and competing views, it is worth emphasizing the importance of the date of the plate. It is generally accepted that iron and steel were not made in this quantity until about 1500 B.C. Certainly, examples exist in that time frame, some of them famous ones. For example, daggers of both gold and iron were found on Tutankhamun's mummy (Fig. 1) which is known to be from 1350 B.C. There are occasional claims that older pieces exist; for example, it is claimed that an iron knife blade in a museum in Turkey is from 2500 B.C., but there is no supporting evidence presented.

Nonetheless, some significant authors have proposed a much older start to the Iron Age. For example, a former President of the United States, Herbert Hoover (1928-1932), was a mining and metallurgical engineer (Stanford University, 1896), who became a famous and wealthy engineer before entering the political scene. He and his wife translated the famous text *De Re Metallica* by Agricola, from the Latin to the English in 1912.[6, p.421] In that book, he footnoted his thoughts on the history of iron in Agricola's section on iron making. He considered that the beginning of the iron age was in the prehistory period, that the Egyptians knew iron 5000 to 6000 years ago, and used iron tools to carve the stones of the great Pyramids. Thus, if the iron plate of Gizeh could be accurately dated, it would be a significant point in determining the evolution of large, man-made, iron-based artifacts.

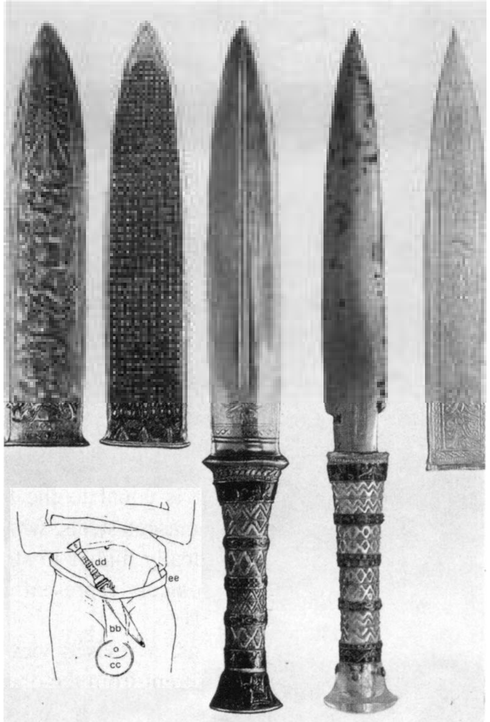

Figure 1. Evidence of steel from 1350 B.C. Daggers of iron and gold from Tutankhamun's grave, and their sheaths. Insert: Position of iron dagger on Tutankhamun's mummy. (After Sherby[7])

In order to resolve the issue of the date of the plate, it is possible to turn to ^{14}C dating. Using this technique, the dating of ancient steels has, in fact, been done successfully. In the last decade in particular, carbon dating on relatively small samples, weighing as little as a fraction of a gram to several grams, has been accomplished by using accelerator mass spectrometry (AMS). One of the best AMS machines is at the Lawrence Livermore Laboratory. The possibility of establishing a capability to age steel and iron objects is being explored by one of the authors and a colleague.[8]

Cresswell[9] in 1992 published a good summary of the history, issues, and limitations surrounding carbon dating of iron and steel artifacts. In summary, Turekian first conceived the use of ^{14}C dating for artifacts and van der Merwe built a system at Yale to do so in the early 1960s. The system required 500 mg of carbon equivalent, however, which corresponded to up to 1 kg of iron thereby severely restricting the use of the technique. Developments in the 1970-1990 period succeeded in reducing sample size, but only to the level of tens of grams. The transition from proportional counters to the

AMS technique of Cresswell[10] in 1991 allowed reductions in carbon equivalent to 5 mg. The sample size (which of necessity depends upon C content of the steel) ranges from about 5 g for wrought iron (0.05%C) to about 100 mg for cast iron of 2%C content.

For ^{14}C dating to be meaningful, the source of C used in the steel making has to be charcoal or freshly cut wood. Sources such as coal and coke are exhausted of ^{14}C. The dilution or contamination by lime and recycling of artifacts also has to be considered. Only in the 19th century did coke become a universal fuel in the industrial world. In fact, up until the Industrial Revolution, most smelting was carried out using charcoal-fired furnaces and historical records indicate that freshly cut wood was a fuel source. So extensive was the use of charcoal that vast deforestation took place in the US in Pennsylvania in the 17th and 18th century. In England, an act was passed by Queen Elizabeth I in 1558 restricting the use of timber for fueling iron smelts. It is worth noting, however, that the Romans and the Chinese from the 4th century A.D. did use coal.

Cresswell points out that meteoric iron, or even terrestrial iron, can also be incorporated in ancient steel making and these forms can have quite high carbon contents (up to 2.5% in both combined and graphitic forms). Although these contributions could confuse the dating of ancient artifacts, the presence of meteorite iron can be identified by its high Ni content (4 to 7%) whereas terrestrial iron is rare and is sufficiently well-documented to not be problematical. Other complications arising from the use of coal can sometimes be indirectly determined.

Table 1. A summary of ancient iron and steel artifacts dated using the ^{14}C technique by accelerator mass spectrometry (* smallest sample dated)

Object	%C	Grams	Age, BP	Reference
Iron Hook	0.18	4.53	1330±110	Nakamura et al.
Japanese Sword	0.49	2.27	880±150	Nakamura et al.
Frobisher Bloom	0.30	1.34	1340±70	Cresswell
Luriston Dagger	0.30-1.0	0.485	2940±60	Cresswell
MIT Dagger	0.30-1.0	1.44	2880±60	Cresswell
Sri Lanken Wootz*	1.79	0.274	980±40	Cresswell
Cast Iron Planing Adze	3.6	0.93	1770±160	Nakamura et al.

Wootz steel from Sri Lanka was analyzed by Cresswell who describes it as "ideal for ^{14}C dating"; the sample size required was a mere 274 mg because of the high C content of the wootz (1.79%C).

Subsequent to the work of Cresswell, other studies have appeared. For example, Nakamura, et al.[11] dated a Japanese sword (2.27 g, 0.49%C), a planing adze (0.93 g, 3.6%C), and an iron hook (4.53 g, 0.18%C). Table 1 summarizes the dating of steel artifacts using AMS.

Thus, it is concluded that it should be possible to date the iron plate at Gizeh using modern AMS ^{14}C techniques.

Achilles Shield: Perhaps the earliest known reference to improved properties in a laminated metal composite can be found in *The Iliad of Homer*[12] in 800 B.C. which describes Achilles' shield as having five layers — 2 bronze, 2 tin, and one gold. The laminate was in the sequence bronze/tin/gold/tin/bronze. During combat, the superior performance of the laminate was demonstrated by the fact that Aeneas' bronze spear penetrated the first two layers but stuck in the gold layer. Some details of the encounter between Aeneas and Achilles can be found in translations of Homer as shown below:

> *But from battle, seeing I am eager therefor, shalt thou not by words turn me till we have fought with the bronze man to man; nay, come, let us forthwith make trial each of the other with bronze-tipped spears.*
>
> *He spake, and let drive his mighty spear against the other's dread and wondrous shield, and loud rang the shield about the spear-point. And the son of Peleus held the shield from him with his stout hand, being seized with dread; for he deemed that the far-shadowing spear of great-hearted Aeneas would lightly pierce it through – fool that he was, nor knew in his mind and heart that not easy are the glorious gifts of the gods for mortal men to master or that they give place withal. Nor did the mighty spear of wise-hearted Aeneas then break through the shield, for the gold stayed it, the gift of the god. Howbeit through two folds he drave it, yet were there still three, for five layers had the crook-foot god welded, two of bronze, and two within of tin, and one of gold, in which the spear of ash was stayed.[12]*

Achilles then turns his spear upon Aeneas' shield, but that shield also protects Aeneas. The contest turns to swords and stones. Poseidon intervenes when he realizes Achilles and Aeneas will kill each other, and spirits Aeneas away while shedding a "mist over the eyes of Achilles." It is of interest to note that steel was known and described in the Iliad, but was not used in the description of a tough laminated material. For example, as the battle subsequently continues, Hector of the Trojans goes against Achilles but fears "his fury as the flashing steel." Later, a description of the hardening of steel by quenching is given. Also, in the 33rd book of the Iliad, Achilles offers a lump of iron as a valuable prize at the funeral games of Patrochus, although there is the possibility that this is meteoric iron.

Adze Blade: The solid state joining of two dissimilar ferrous materials is well documented as being practiced as early as the first millennium B.C.[13] The welded product often consisted of a steel and an iron. A photomicrograph is shown in Fig. 2, of an adze blade (a cutting tool used in farming), made by Greek blacksmiths around 400 B.C. The figure shows a fairly sharp interface between a carburized iron cutting blade adjoining a low carbon backing plate. The blade was found at Al Mina, the ruins of a Greek trading colony on the coast of Turkey near Syria. The motive for using a sheet of carburized iron for the working face of the adze, but soft iron for the other face, was an economical one based on the scarcity of carburized iron.

It is worth noting that in about this era, it is believed that Alexander the Great was given a gift of the India steel, "wootz" by the Indian King, Peru. The wootz, which was contained in a gold box, was the starting material for the famous Damascus steels. The patterns on Damascus steel arise from the aggregation of spheroidized, proeutectoid, cementite stringers as a result of the specialized processing of the ultrahigh carbon content (1.3 to 1.8%C) steels. However, it is quite likely that in some cases, laminated composites were developed in an attempt to duplicate the Damascus steel pattern. This is because such patterns certainly could appear to be the result of the intimate mixing of two dissimilar metals. Damascus steels were famous through the centuries. Their use in the Crusades by Saladin, the leader of the Saracen warriors, in a meeting with Richard the Lionhearted, was immortalized by Sir Walter Scott in his book (1871) *The Talisman* which was subsequently made into two movies and a BBC television mini-series, thereby bringing the fame of these steels into the modern times.

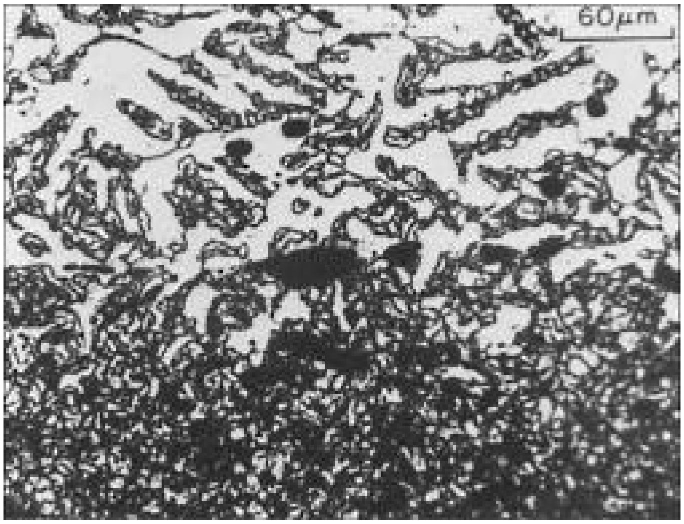

Figure 2. Shown in the above figure is an Adze blade, which was made in about 400 B.C., found at Al Mina on the coast of Turkey. The cutting edge is medium carbon steel and the backing piece is a low carbon steel. The authors thank Dr. R. Maddin for permission to publish the above photograph.[14]

Chinese Steel of Hundred Refinings: Rubin describes early iron making in China in a recent paper.[15] He examined over 1000 iron artifacts from 60 iron making sites and tombs. The artifacts dated from between 900 B.C. to 1800 A.D. Of interest to the present paper is his discussion of "steel of hundred refinings." The phrase "hundred refinings make quality steel" is a Chinese saying dating to the 2nd century A.D..

In examination of a knife of "thirty refinings", from 112 A.D., Rubin notes that the knife:

> *...seemed to be a composite of approximately 30-36 layers. The suggestion was proposed that the number of refinings specified the number of layers after repeated doubling. Thus 30 and 100 refining would probably indicate 32 and 128 layers respectively and sword[s] of 60 or 50 refinings (actually 64 layers) should be expected.*
>
> *This was confirmed by an excavation soon after the prediction was made. A sword of marked 50 refinings dated back to 77 A.D. was unearthed from a tomb in Xuzhou, Jiang-su....Examination showed that...it consisted of alternative low and high carbon layers, about sixty in total. The cost of the sword was also marked as worthy of 1500 coins, equivalent to grains enough for one man's two and half years' living.*

Merovingian Pattern-Welded Blade: An early European technique dating from about the end of the second century resulted in the Merovingian pattern-welded blades. (Iron objects manufactured prior to this date are frequently too severely corroded for their surfaces to be evaluated metallurgically.) According to Smith[16], Merovingian blades were primarily manufactured on the Rhine although they were widespread through trade and war. The blades consisted of strips of pure iron and carbon steel (or iron strips that had been carburized on one side) hammered or forged together in a manner involving folding or twisting. The cutting edge consisted of the high-carbon-content steel, often inserted between plates of low-carbon material. Upon grinding to shape after heat treating, patterns arising from the different layers become visible. In addition, it is quite likely that etching in fruit juice or sour beer was carried out to develop the patterns. An example is given in Fig. 3, from a blade discovered in a Viking grave in South Finland.[17]

Thus, beginning in the period around 500 A.D., pattern welded daggers and swords were made, including Viking blades starting in about 600 A.D.. Smith[16] comments that the edges are martensitic between plates of iron in a manner similar to the Japanese sword. He also notes that it is "difficult to justify the particular pattern used in the center of the swords on any but aesthetic grounds." Included in this group of materials is the early Japanese sword.

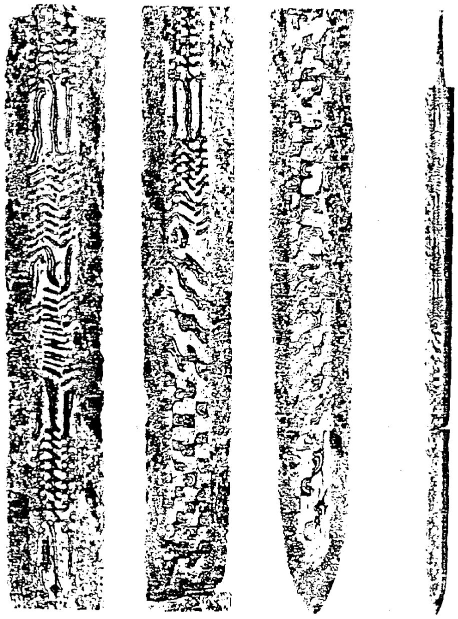

Figure 3. Merovingian pattern-welded blade discovered in a Viking grave in the South of Finland. It was most likely made on the Rhine in the period 650-700 A.D. Helsinki University. Courtesy of *The Gun Report*.[18]

Japanese Sword: The Japanese sword has universally been regarded by eminent metallurgists as the ultimate expression of metallurgical accomplishment. For example, in 1962, Edgar C. Bain[19] wrote:

> *The old swords of Japan are probably the best examples of the almost incredible pains taken to produce a superb implement.*

Also, in 1960, Sir Cyril Stanley Smith[16] wrote that in his opinion:

> *The Japanese sword blade is the supreme metallurgical art.*

Certainly, world wide, the Japanese sword (*Nippon-tô*) is one of the most famous of all swords. The sword has always held a special place in the history and culture of Japan. Japanese legend, for example, tells us that Susanoo, brother of the Sun Goddess Amaterasu, slew an eight-headed dragon with a single stroke of a sword. Despite this, the blade was deemed to be inferior and Susanoo was given a magnificent sword from the dragon's tail. He gave the sword to his sister, the Sun Goddess, who then handed to her grandson the Imperial Regalia which included three items — the jewel, the mirror, and the sword.

From a metallurgical viewpoint, the Japanese sword is of interest at several different levels. First, their manufacture involved the solid-state bonding of steels to themselves and to steels of radically different carbon contents. That is, in its simplest form, with the exception of the very earliest blades, the sword is a composite. An example of one of the composite designs is that of a high-carbon external sheath surrounding a low-carbon core. The procedure used to make such a blade is shown in Fig. 4. This composite approach allowed the swordmaker to achieve the desirable properties of both hardness and ductility in a single weapon.* Often, one of these properties is only developed at the expense of the other.

Second, to produce the high-carbon part of the blade, the steel was subjected to multiple folding operations; this has given rise to the erroneous concept that the swords contain millions of discrete layers. This point is experimentally examined in the next section on Modern Laminated Metal Composites.

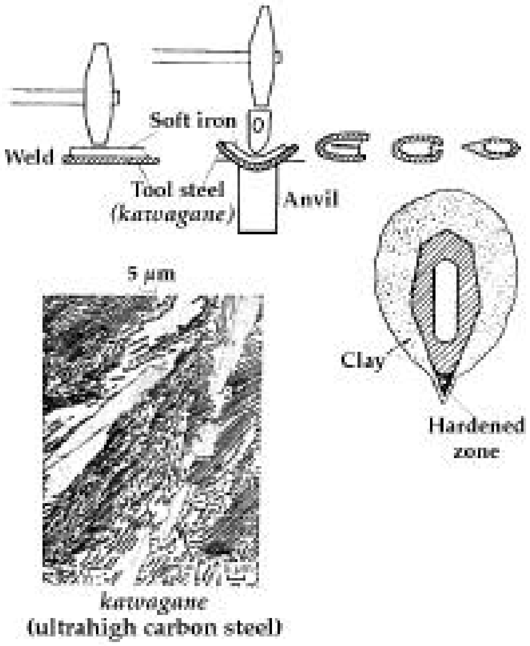

Figure 4. Procedure used by Japanese blacksmiths to make laminated tools by solid-state bonding ultrahigh carbon steel, known as kawagane, to soft iron; including a cross section of a blade coated with various thicknesses of clay to control temperature, with minimal clay coat at the edge to allow proper hardening. (adapted from E. C. Bain [19])[14]

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* Tylecote[20] has pointed out a similarity in this regard between the Japanese sword composite design and the much later development of "shear steel" in Western Europe following the Industrial Revolution. The similarity lies in the fact that comparatively few pieces of steel of different carbon content were welded together to make a composite, single-edged, blade which was finally heat treated. In shear steel, mild steel outer strips encased a high-carbon center strip — a design currently available in hand-made knives by contemporary artisans. In fact, Tylecote goes as far as to say, "There is essentially no difference in principle between a scythe [made from shear steel], and a Japanese sword."

Third, the development of the complex and beautiful surface markings was a consequence of a selective surface heat-treatment process (known as *yaki-ire*), achieved in part by covering the blade with different thicknesses of clay; this gave rise to an intriguing combination of transformation products and surface patterns. This was not only a visible and aesthetic example of the skill of the swordmaker, but it was also evidence that the edge of the blade had been hardened.

The blade must have a recognizable pattern, called the *hamon*, where the structure changes from the hard martensite at the edge, called *yakiba*, to soft pearlite. The *hamon* is perhaps the most important aesthetic feature of a blade, and the first thing sword aficionados look for, as it is essentially the swordsmith's signature. Kapp et al.[21] cite the work of B. W. Robinson's classic book *The Arts of the Japanese Sword*[22], illustrating fifty-three different *hamon*, each with its own name (from the descriptive "straight irregular" to the more suggestive "chrysanthemum and water") and the name of the smith or school with which it is identified. (See Fig. 5 for an illustration of broad classes of *hamon* patterns.)

The Japanese sword's external sheath has a similar carbon content (i.e., it can be hypereutectoid, about 0.8-1%C) to that of the Damascus sword (also hypereutectoid, but in the 1.5-1.8%C range). This similarity of composition is especially so in the early stage of manufacture of the Japanese sword in which steel of about 1.8%C is used.

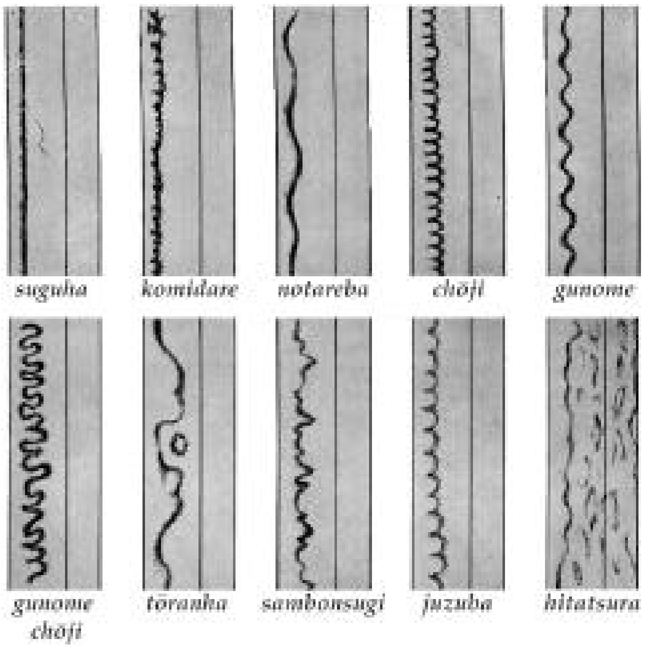

Figure 5. Types of Hamon. (After Sato [23])

The high-carbon steel (called *kawagane*, but also sometimes called *uagane*) used for blademaking was principally prepared using a reduction process method, in which iron, sand, and charcoal produced *tama-hagane*. A fixed amount of iron ore and charcoal was mixed and heated in air to 1200°C, resulting in products of molten pig iron, slag, and unmelted ultra-high-carbon steel. The iron ore came from so-called black sands known as *satetsu* (iron oxide). The carbon was added to the black sand in a smelter called a *tatara*. When the pig iron and slag were allowed to separate by pouring, the end product was lumps of ultra-high-carbon steel containing about 1.7%C (*tama-hagane*). This material was then repeatedly forged and folded until the appropriate shape and reduction in carbon content was achieved through decarburization. (It should be noted that, depending upon the carbon additions, temperature, and time at temperature, the result of such a repeated folding and forging process <u>could</u> be low-carbon steel.)

Forging the blade involved a number of steps. First, the *tama-hagane* was repeatedly forged and folded to produce the *kawagane* which becomes the sheath or jacket steel. Second, the *shingane*, or low-carbon core steel, was formed, also by a repeated folding procedure. Third, the low-carbon core was inserted, by one of several methods, inside the high-carbon jacket steel. In the fourth step, the composite was drawn out to the approximate length of the blade; and the fifth step shaped the final blade.

The end product is a *kawagane* steel with excellent mechanical properties because the carbon content is both relatively low (about 0.6-1.0%C), and the carbides are distributed uniformly in a fine-grained matrix. As discussed later, no visible pattern-welded structure is obtained from this scale of folding, not only because the individual, 0.2 μm layers are unresolvable to the naked eye, but also because the carbon content of each layer is identical (carbon atoms diffuse a distance of 1.4 μm in 30s at 1000°C). An observable pattern-welded structure, however, often emerges from the final several folds.

Thus, the method of manufacture and the origins of the surface patterns on the Japanese sword are quite different from those of Damascus swords. Specifically, the principal surface pattern on a Japanese sword is created as a result of the various transformation products following heat treatment of the blade. There are also surface patterns that consist of a gross texture from the final stages of piling, folding, and forging. The earliest reference to surface patterns on a Japanese sword, referenced by Smith[16], is to 1065 A.D.. There are subtleties to these patterns that illustrate several intriguing metallurgical issues.

There are an estimated one million swords now known to exist; 117 have been designated as Japanese national treasures. The most famous and revered of the swords are identified with a name or *meito*. Two such examples are the *ō-kanehira* by Kanehira and the *dojigiri* by Yasutsuna made over 900 years ago. Photographs of the *ō-kanehira* are shown in Fig. 6.

Figure 6. *O-Kanehira*. *Tachi* by Kanehira. Steel. *Nagasa* 89.2cm. Mid-Heian period, approximately 1000 A.D.. Tokyo National Museum. Signed *Bizen no kuni Kanehira* ("Kanehira of Bizen providence"). (After Sato [23])

Medieval Damascened Knives: Piaskowski[24] has reviewed in detail pattern-welded damascened knives found in Poland dating from the 8th to 12th century. Their real origin is not clear, but Piaskowski believes that some of the knives may be examples of the work of early medieval Polish smiths. The knives all consisted of three regions: a steel cutting edge, adjacent to a complex, central, patterned layered region of carburized iron and iron, and a backing layer of iron or steel. This is in contrast to other related techniques in which a steel central layer is sandwiched between patterned layers of iron and steel. Evidence for heat treatment to produce martensitic structures, and even tempered structures, is described. Techniques involving from 3 to 17 initial layers are presented. In all cases, hammer welding and plastic deformation took place during manufacture. The carbon contents of the layers are poorly evaluated, however, and only one composition, 0.5%C, is given for one steel in one of the knives.

Thailand Axes and Tools: Although less extensively developed than the Indonesian metal krisses (next section), there are interesting artifacts originating in Thailand. In a study of such objects,[25] four iron artifacts were excavated in Northeast Thailand and three were dated to the Late Iron Age (e.g. 300-400 A.D.). The fourth artifact, dated by association with the other three, involved welding an ultrahigh carbon content steel onto a wrought iron core to form a high quality axe blade. In concluding that the artifacts date to the Late Iron Age, the authors note that iron was produced in Thailand from 500 B.C. or earlier.[26]

The crescent-bladed axe was found a short distance from the Late Iron Age mound site of Non Phrik which is located near Ban Hua Na village, Phu Luong sub-district, Loei Province. Although the carbon content was estimated as 1.8% carbon and the steel was concluded to be hypereutectoid, this finding was based on the assumption that the grain boundary material was all massive carbide. It is not clear to the present authors that this is the case at all, and the steel may in fact be hypoeutectoid. Nonetheless, the artifact is an example of an axe that has a relatively massive cutting head formed by welding a layer of carbon steel onto a wrought iron core.

In the microstructure of a second artifact, an iron socketted chisel, a “laminated semi-circular pattern is readily visible.” Hogan and Rutnin[25] proposed that the manufacturing process was “a simple procedure”.

> *The starting material was piled wrought iron, made by hammering sponge from the smelting furnace into thin sheets, then folding and re-folding while hot and hammering them together to form a shape required for sale to blacksmiths. When re-heated in the forge the surface of each sheet may be either oxidised or reduced, so that the carbon content is different in surface and centre of the sheets. When these sheets are welded together the laminated appearance results.*

Laminations were also evident in an iron socketted spearhead which:

> *...appears to have been made in a sandwich construction, commencing with a relatively high carbon strip of the iron, lying parallel to the top surface.... This was sandwiched between strips of soft, low carbon iron and the sandwich forge welded and shaped....*

Thus, the:

> *...use of a higher carbon core ensures that the point of the spear can always be resharpened to give a relatively hard, sharp point, while the soft outer layers are more easily ground to remove the bulk of the metal required for sharpening. The higher carbon central strip would have been prepared by the smith by cementation, ie [sic] by burying a thin strip of low carbon iron about 1 mm thick in a charcoal fire with limited air access to give a reducing atmosphere, rich in carbon monoxide.*

Indonesian Kris: The Indonesians of Java and other Malayan Islands made a number of knives known as krisses. Indonesian krisses usually are forged to have repetitive curves along their length. There are in fact two classes. One is that containing long blades that were used as sabers, with a slashing motion. The other class is that of short stabbing blades. All are double edged. It is believed that the undulating curves might make for more efficient thrusts and recoveries of the weapon. Other theories are based on religion. For example, under the Hindu influence, the oriental snake gad, Naga, may be represented in the serpentine curves. Unlike Japanese swords, the krisses bear no names, dates, or places of manufacture, although there are over 30 different types that can be associated with different regions. The blades are usually laminated; in fact, the name for the most popular ones is *pamur*, a Malay word for combination or mixture. Smith[16] included excellent examples of surface patterns in his book on historical metallurgy; and the detailed manufacture of a relatively modern kris also has been described by the famous metallurgist Walter Rosenhain.[27]

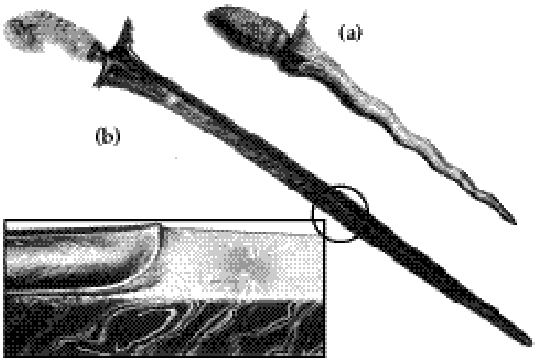

Figure 7. (a) Typical Indonesian kris. (b) Indonesian executioner's kris is a composite of meteoric nickel iron and plain carbon steel.

A typical Indonesian kris is shown in Fig. 7(a), and an interesting example of a specialized executioner's kris, with a straight blade, is shown in Fig. 7(b). In this case, as with others, one of the layers is meteoric iron containing Ni. According to Smith, krisses were made from about 1379 A.D. onward in Indonesia under Hindu influences. From the description by Rosenhain, the modern kris was made by solid-state welding of a tool steel ("a 'high carbon steel' such as is commonly used for tools and cutlery," to quote Rosenhain) to welded layers of wrought iron. In addition, according to Rosenhain[27]:

> *The imperfection of the [solid-state] welds between the wrought iron [layers] also play an important part in the formation of the damask pattern.*

Halberds: A halberd is a weapon that is both a spear and a battle ax that was used in warfare in the 15th to 16th century. According to Meier[28], statements regarding the distribution and origin of the halberd are rarely met with until the 15th century. But, poems, songs, and chronicles provide written allusions to the halberd from the 13th century. Also, because of the troop and arms-rolls records of Zürich from the time of the Old Zürich War, some firm assertions can, in fact, be made with regard to the distribution of the halberd. For example, in 1442 A.D., of the 1,591 short-arms counted, 856 were described as halberds.

The halberd of the 14th and 15th centuries was conceived principally as a cutting weapon; the halberd was fixed on the ash shaft with sockets (straps). The construction of the blade of the halberd was improved in the course of the 16th century, thanks to better raw materials and new forging-techniques. Until about 1500, the blade of the halberd was generally composed of four pieces. In the 16th century the cut and thrust function of the halberd acquired importance. Henceforth the point of the weapon lay in the axis of the shaft. The centrally mounted halberd blade which was attached to the staff with shaft-straps, comprises ten pieces, as for example on a "Sempach-halberd" of the 17th century. It is of interest to note that the nature of the construction parallels that of other laminated weapons in that a high carbon steel cutting edge is surrounded by a low carbon sheath.

Chinese Blades: Chinese steel of "hundred refinings" from 100 A.D. was mentioned earlier. Other relatively recent examples of welded knives and swords exist from Chinese blacksmiths. In addition to perhaps being the originators of the Japanese sword in the 5th century A.D., they also made their own pattern welded blades as shown in a 17th century example in Fig. 8.

Figure 8. A 17th century Chinese sword of the pattern-welded variety. The pattern shown on the macrograph arises from the difference in etching of the two dissimilar steels from which the sword was manufactured, involving a sold-state bonding process. Courtesy of the Metropolitan Museum of Art, New York. [17]

Pattern Welded Blades of More Recent Origin: A more recent example, i.e. a welded Damascus steel dagger, made about 150 years ago, is shown in Fig. 9(a) and illustrates the multi-layered composite nature of the weapon. A low magnification picture of the dagger, shown in Fig. 9(b), illustrates the unique surface markings. These markings were created by forging many alternating layers of high carbon steel and iron plates, followed by a complicated twisting and forging step. The micrograph in Fig. 9(c), taken at high magnification, readily identifies the layers as consisting of alternating high carbon spheroidized steel with graphite stringers and low carbon iron layers.

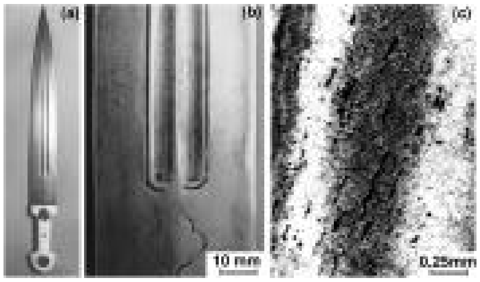

Figure 9. Welded Damascus steel dagger. (a) dagger; (b) unique surface markings, low magnification; (c) micrograph showing distinct layers of high carbon spheroidized steel (dark) and low carbon steel (light).[1]

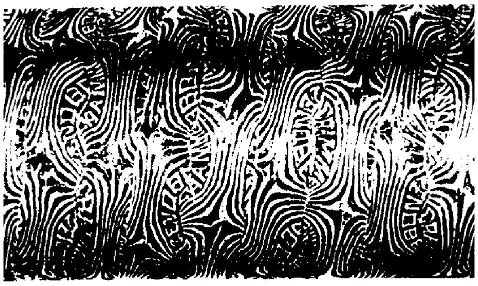

Figure 10. Welded Damascus gun barrel with the words ZENOBE GRAMME worked into the pattern. This advanced stage of development was reached in the 19th century. Musée d'Armes de Liège. (After Puraye [29]) [17]

European Welded Damascus: After its origination in the East in the 16th century, a technique of welding steel to iron, in strips, to give strength and texture to guns and swords, was pursued in Europe from the end of the 18th century. A typical welded Damascus barrel would have consisted of seven layers (four of low carbon material or pure iron, and three of steel). These would have been forged together, and then the resulting strip coiled to the desired barrel shape. In more complex routes, the welded strip was twisted, and then several such strips were re-welded and re-forged repeatedly to provide intricate final patterns. Some swords were made in this manner (for example, the welded Chinese sword shown in Fig. 8) and, in some cases, were attempts to duplicate the true Damascus steels.

Interest in welded Damascus steel was promoted in France in the early 19th century. High levels of skill were developed so that inscriptions made of one steel could be worked into another steel; an example is given in Fig. 10. In some cases in Kashmir and Constantinople the patterns arose from differences in the oxidized surfaces of the steel strips.[16] It should be noted that contemporary advice is that such welded Damascus shotguns may be unsafe to fire, especially using modern powders.[30]

Modern Welded Damascus Objects: In addition to their historical significance, pattern welded knives are currently being made by blacksmiths the world over. (A companion paper in this conference discusses these laminates.) The major objective of these modern welded [Damascus] steels is to create an artistically beautiful object. The mechanical properties are not usually considered to be an important factor since the product is not specifically utilized for structural applications. For example, in a contemporary article on pattern-welded Damascus blades, Warner[31] gives a good summary of the benefits and disadvantages of that class of knives.

> *The principal reason for Damascus blades is that most people consider them prettier than plain blades. This beauty is in the nature of the blade and not applied to its surface, which is the fascination for the makers. Another reason Damascus attracts is that it is very difficult to make. There is a lot of craft involved, and it shows. And still another reason for Damascus steel is that it can make a superior knife. It does not always do so, but it can.*

Examples of American welded Damascus steel knives are shown in Fig. 11. Typically there is a long waiting list for purchasing such knives from the most famous of the knife makers. As discussed in the companion paper in this volume, there are at least 1000 custom knife makers, and an overwhelming number of styles of knives. In this compendium, there are no less than 38 different types of welded Damascus blades including knives advertised as having from 64 to 1000 layers, various combinations of alloys and steels, and a number named after specific blacksmiths.

Figure 11. Welded Damascus steel knives made by American blacksmiths Bill Moran (top, courtesy of W. Moran) and Devin Thomas (bottom, after Thomas[32]).

In Japan, knives and scissors with similar surface markings are made by a mass production process. Three-

Table 2. Some Examples of Ancient Laminated Composites

Material	Approximate Era	Composition (where known)	
		Layer A	**Layer B**
Gizeh Pyramid Laminated Steel Plate	2750 B.C.	low carbon steel ~0.2%C	wrought iron
Achilles' Shield	700-800 B.C.	5-layer composite of bronze/tin/gold/tin/bronze	
Adze Blade (Turkey)	400 B.C.	medium carbon steel ~ 0.4%C	low carbon backing plate ~0.1%C
Chinese Blade "Hundred Refinings"	100 A.D. onward	negligible	low carbon
Merovingian Blade	2nd - 12th century A.D.	carbon steel	"pure" iron
Japanese Sword	400-500 A.D. to present		
Overall Blade		outer sheath: 0.6 - 1.0%C	inner core: 0 - 0.2%C
Outer Sheath — Initial to Final Condition		1.6%C reduced during 12-20 foldings (see text) to 0.6-1.0%C	interlayer regions during final foldings may be low in C due to decarburization
Thailand Tools	400-500 A.D.	negligible	0.13, 1.8(?)%C
Indonesian Kris	14th century A.D. onward	tool steel ~1%C	low carbon; meteoric iron (Fe – 5-7%Ni)
Halberd	14th century A.D. onward	high carbon	low carbon (complex assembly)
Chinese Pattern Welded Blade	17th century A.D.	unknown	unknown
Shear Steel and Double Shear Steel	19th century A.D.	high carbon	mild steel
European Gun Barrels	19th century A.D.	steel ~0.4(?)%C	low carbon steel or pure iron
Persian Dagger	19th century A.D.	~0.8%C	~0.1%C

layer laminated working chisels are also made in Japan. In Norway, a three layer laminate knife, made by solid state brazing, is a popular item. This knife is used by sports and cutlery enthusiasts. Typically, the central layer is a high carbon tool steel and the outside layers are low carbon or stainless steels. In this case, somewhat in contrast to the Japanese sword, but similar to the modern Japanese chisels, the sharp, high hardness edge in the center is inhibited from cracking by the presence of the tough outside layers. In an article on testing woodworking chisels, the following comments were made:

> *The laminated Japanese chisels* we tested were made in a style once found in Virginia in the 18th century. Steel was scarce then, so only a small piece was used for the cutting edge of the chisel. Iron was used for the body because it was less expensive.[34]*

This use of steel in a sandwiched structure of iron because of its relative scarcity is mentioned as a motive for early laminates and continues for other reasons to be the design of some modern laminates.

Finally, some structures that appear to be laminated may not be. For example, coffin nails recovered from burials at a slave cemetery at Catoctin, in Frederick, Maryland, can have a laminated appearance. The nails were shown to be hand wrought. In some cases, smiths did make a nail rod by welding together rods of scrap iron and drawing out a bar. In contrast, in the same era, industrial production of nail rods involved taking 4%C pig iron and repeatedly heating and forging, resulting in the oxidation of the carbon and the formation of a near-pure wrought iron. The microstructures of nails from this approach can appear to be laminated wrought iron surrounding a mild steel core, but in fact are concluded to be a core of incompletely decarburized metal that has survived the heating and forging for converting smelted pig iron to a carbon-free wrought iron.[35]

A summary of the ancient laminated composites is given in Table 2.

Modern Laminated Metal Composites

Historically, as described in the early part of this paper, there are a number of reasons why laminated materials were made. From the viewpoint of modern

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* The Japanese traditionally make chisels by forge welding (often by hand) a hardened, tool-steel blank to a mild-steel billet.[33]

engineering structures, these reasons would have to focus on improvement in mechanical properties or possibly the economical aspect of inserting expensive material between less expensive materials. In the following sections, some examples of modern engineering laminated structures are first described. This is followed by a description of how laminates can be manufactured. Included in this section is a description of an experiment to evaluate the retention of layer discretion in laminates made by multiple folding techniques. In the third section, the mechanical behavior of laminates is discussed — the room temperature tensile properties, the high temperature tensile properties, the impact behavior, and damping properties. The impact behavior in many respects is the most interesting from the viewpoint of enhanced properties in ancient laminated composites.

Examples of Modern Laminated Metal Composites: The practical application of LMCs is far more developed in the Former Soviet Union than in the western world. Contemporary engineering examples of LMC technology include the use of laminated materials for fracture critical applications involving large pipes,[17] large pressure vessels,[36] and gun tubes.[36] The gun tubes consisted of one or more cylinders that were assembled and shrunk-fit together.[36] In all these applications, internal interfaces between layers limit crack propagation through several different mechanisms. In the pipe and pressure vessel applications, thin sheets of steel are tightly wrapped together and welded to form concentric shells that resist through-thickness crack propagation. The pipe application, which was developed at the Paton Welding Institute in the Ukraine, is especially noteworthy, since in addition to the through-thickness toughness obtained from the concentric shells, toughness in the longitudinal direction of the pipe was obtained by introducing periodic circumferencial segments. An example of this product, photographed by one of the authors (JW) during a visit to the Ukraine in 1993, is given in Fig. 12. On the top is a picture of the overall pipe section, and below is a close-up of the thin sheet configuration.

At the Moscow Steel and Alloy Institute, a unique metal forming process has been developed for the manufacturing of concentrically laminated gun tubes.[17] The process (called the "radial shear helical rolling procedure"), which is highly developed in Russia, uses three eccentric rolls that permit large amounts of deformation without change in shape. This process has been used on steels with a wide range in carbon content, including extremely high carbon steels (or, in fact, white cast irons) containing 2.0 and 2.6% carbon. The technology has also been used to manufacture concentrically layered rods for use as high strength track pins in vehicles such as tractors and tanks.[37]

In addition to the steel/steel laminates described above, bimaterial laminates have been manufactured including Al/steel, Cu/steel, and Al/Cu.[17] These bimaterial laminates were manufactured by explosive bonding and welding techniques developed at the Moscow Institute of High Temperature and the Design and Technology Institute of High Rate Hydrodynamics in Novosibirsk. At this latter institute, over 80 combinations of metals have been successfully laminated including some in which multi-layer laminates have been formed. Their main product at present is aluminum (~ 3 mm thick) bonded to steel (~ 20 mm thick) which is then made into large rod bearing caps for ship and train diesel engines. They have bonded Cu to steel in plates 1.5 m x 2 m and claim to have produced laminates containing 50 layers of steel and aluminum. The processing, quality control, and industrial production of LMCs have been reviewed in a textbook by Potapov et al.[38] The materials discussed are intended primarily for applications requiring good corrosion resistance characteristics and wear resistance.

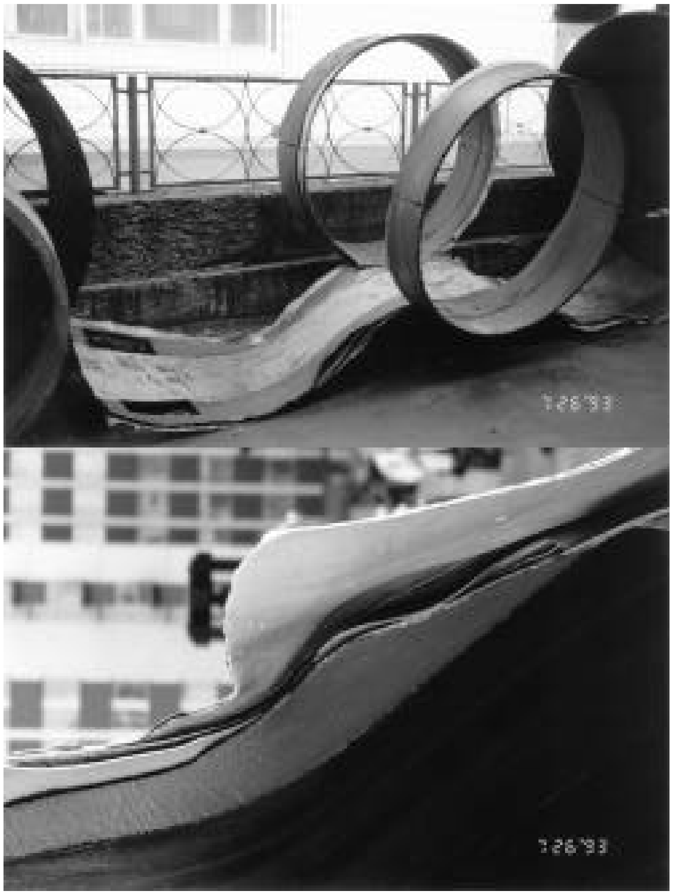


Figure 12. Large pipe manufactured in the Ukraine.
Top: Overall view.
Bottom: Sheet configuration.

Manufacturing of Modern Laminated Metal Composites: Modern laminated metal composites can be made by many techniques, e.g. bonding, deposition, and spray forming.

Bonding techniques may be classified into several subgroups, such as adhesive bonding, melt bonding, infiltration, diffusion bonding, reaction bonding, and deformation bonding. Surface preparations of the component materials, bonding temperature and pressure, inter-diffusion, and chemical reactions between the component materials greatly influence the micro-structure, chemistry, and bond strength at the interfaces, and overall physical and mechanical properties of the resulting laminates.

Deposition techniques involve atomic or molecular scale transport of the component materials such as in sputtering, evaporation, chemical or physical vapor deposition (CVD or PVD), or electroplating. With the exception of plating techniques or spray deposition, many of the deposition methods are probably too slow and costly to be practical for making large scale, load-bearing components. Barbee[39], however, has used sputtering to make laminates several hundred micrometers thick, containing Cu and Monel layers with individual layer thicknesses of a few nanometers. The materials produced contained many tens of thousands of discrete, individual layers, each of which is only a few atoms thick.

Obviously, ancient techniques for laminations involved folding and mechanical processing. In a modern study designed to simulate part of such ancient methods, some aspects of the multiple folding process, analogous to the process for making *kawagane* from *tama-hagane*, were investigated by devising an experiment which optimized the retention of a laminated microstructure.[40] The laminated microstructure was designed to represent the accumulated, multi-layered composite structure that could result from the development of decarburized surface layers in *tama-hagane*, or shear steel, in which the layers are folded back upon each other during the processing. This modern experimental technique consisted of roll-bonding alternating layers of an ultra-high-carbon steel and an Fe-3Si alloy at a relatively low temperature of 700°C. The resulting laminate was cut into several sections, the sections were stacked, and the stack then rolled. This procedure was repeated until the desired lamina thickness was achieved. The optical photomicrographs (x100) at the top of Fig. 13 show the laminate after three stages: 25, 250, and 2500 layers.

The SEM photomicrograph at the left of the bottom of Fig. 13 is of the 2500 layer laminate and reveals an individual layer thickness of 5 μm. The sample shows a very distinctive separation of the two components, despite the extensive thermo-mechanical deformation. This is only possible because of the special processing conditions and layer compositions that were selected. First, the choice of a working temperature below the A_1 (727°C) prevented dissolution of carbides. Second, the silicon in Fe-3Si inhibited diffusion of carbon from the ultrahigh carbon steel (UHCS).

The other two SEM photomicrographs at the bottom of Fig. 13 illustrate what happened when the 2500 layer laminate was further rolled at 700°C. When a layer thickness of less than 1 μm was achieved (right), the laminate revealed a homogeneous distribution of carbides with a grain size several times the lamina thickness.

These results support the contention that multiple folding of the type used to make *kawagane* steel for Japanese swords in which up to 20 foldings occur, must have led to a homogeneous microstructure at this stage, i.e., processing of the *tama-hagane*. This is especially likely to have been the case because, in practice, the high temperature of folding (1000°C minimum) and the lack of a diffusion inhibitor (such as silicon) in the decarburized layers would have resulted in homogeneous structures much more readily than in our modern example. The final few foldings, however, could have led to a layered effect.

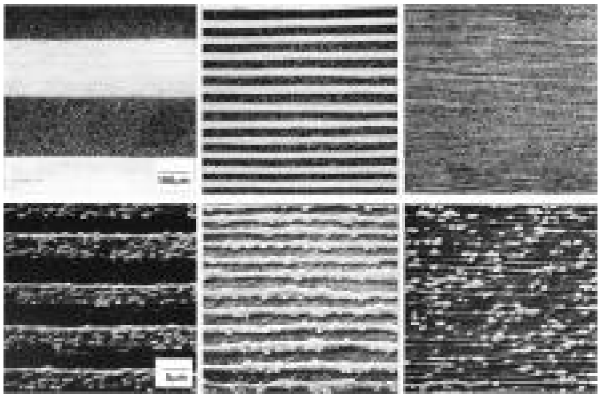


Figure 13. Top: Light microscope photomicrographs of laminated composite of UHC steel/Fe–3Si alloy after three processing stages of (left to right) 25, 250, and 2500 individual layers. Bottom: SEM photomicrographs of the 2500 layer composite processed so as to have individual layer thicknesses of (left to right) 5, 2, and 1 μm. Note homogeneous distribution of carbides in the 1 μm sample. [40]

Tensile Behavior at Low Temperatures: The tensile properties of laminated metal composites have been studied with laminates processed by both the deposition and the bonding method. The deposition method typically results in the creation of ultrafine laminate spacings (from 1 to 0.0015 μm). As seen in the last section, it is not possible to retain this type of layer discretion by multiple folding, and so the benefits of ultrafine layers on strength would not be accessible in ancient artifacts. The second method involving solid state bonding approaches, by rolling or pressing procedures, results in layer thicknesses that are much larger, typically from 50 to 1000 μm.

Tensile Properties of Ultrathin Layer, Laminated Metal Composites: Ultrathin layer composites are usually prepared as foil samples, and typically, only breaking strength is documented. A summary graph of tensile strength data obtained on such materials processed by electro-deposition or by sputtering is given in Fig. 14. The data are for copper layered with nickel or Monel. The figure shows the breaking strength (essentially equivalent to the ultimate tensile strength) as a function of the reciprocal square root of the multilayer periodicity width (also referred to as modulation width). For each set of data, a Hall—Petch type relation is observed, i.e. a linear relation is observed over a range of laminate layer spacing, the strength increasing with a decrease in the modulation width. The trend would suggest that the barrier spacing is an important variable in controlling the strength of the laminate. It is to be noted, however, that a maximum strength is observed for two of the individual investigations; and beyond this maximum, the strength decreases with further decreases in modulation width.

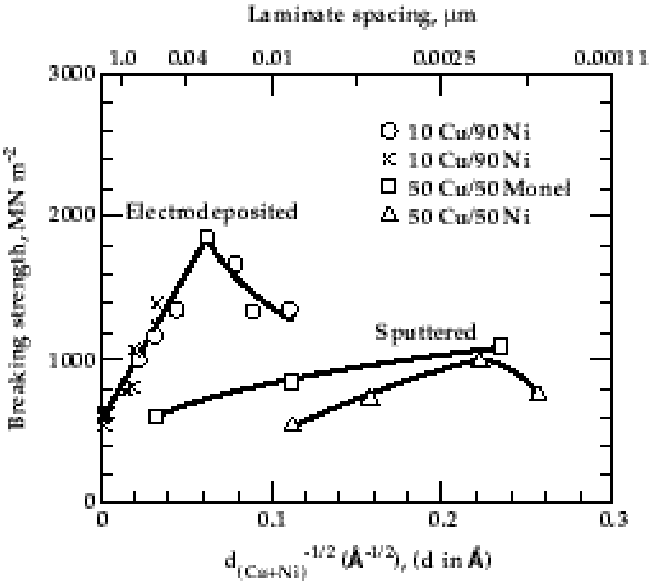


Figure 14. Breaking strength of ultrathin layer laminated composites as function of modulated spacing d (as $d^{-1/2}$). Laminates are based on layers of Cu alternating with either Ni or Monel layers. [1]

Tensile Properties of Thick Layer Laminated Composites: Thick layer metal base laminated composites are usually prepared by solid state bonding procedures. The bonding step commonly involves mechanical working by pressing, forging, rolling, or extrusion.

The tensile yield strength of thick layer laminated metal composites can be readily predicted by the rule of averages. This has been shown for metal systems where two components of equal volume fraction have been investigated. For these cases, the rule of averages is described by the relation:

$$\sigma_y / (\sigma_y)_A = 0.5 + 0.5 (\sigma_y)_R / (\sigma_y)_A \qquad (1)$$

where σ_y is the tensile strength of the composite, $(\sigma_y)_A$ the yield strength of the strong component, and $(\sigma_y)_R$ the yield strength of the weak component.

Fig. 15 shows the application of equation (1) for predicting the normalized strength of the laminate (normalized by the stronger component) as a function of the yield strength ratio of the component materials. The relation predicts the straight line shown in the figure. Also shown are experimental data for metal laminated composites based on aluminum and on ultrahigh carbon steel.[41-46] The laminates shown in Fig. 15 have a wide range in relative strengths — from systems in which there is a larger difference in strength (such as UHCS/brass and Al metal matrix composite (MMC)/Al-5182) to systems in which the yield strengths are very similar (such as UHCS/warm-rolled 304 stainless steel). As can be seen, the data for all laminated composites fit very well with the normalized strengths predicted by the rule of averages.

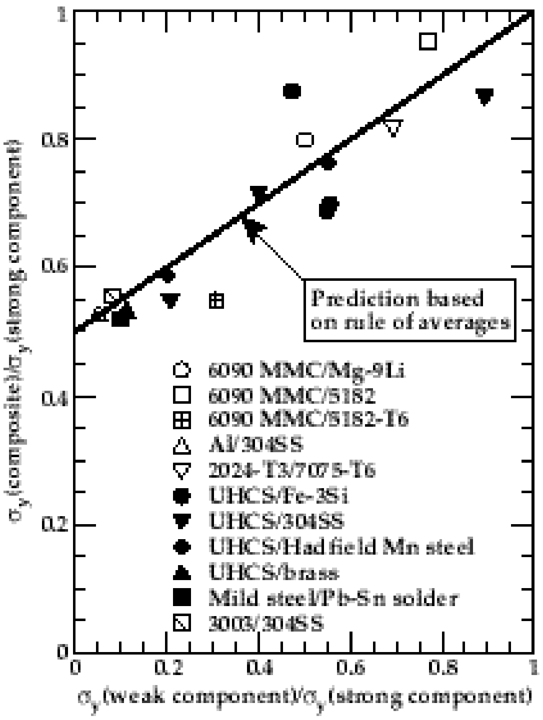


Figure 15. Experimental yield strength of thick layer laminated composites containing 50 volume percent of each component, compared with prediction based on rule of averages (given by the solid line). [1]

Thus, the strengths of laminated ancient artifacts can be estimated with some confidence if the component strengths are known.

The tensile ductility of laminated composites, on the other hand, cannot be predicted by the rule of averages. This is because the tensile ductility of laminates is dependent on many variables, including the susceptibility of the less ductile layer to cracking, the contribution to cracking from the interlayer region, the ease of delamination, and most importantly, the influence of layer thickness.

Fig. 16 illustrates the tensile ductility of laminated composites (the same ones shown in Fig. 15 for tensile yield strength) and compares the experimental results with the prediction for the rule of averages. It is to be noted that the tensile ductility of most of the laminated composites is lower than that predicted from the rule of averages when the difference between ductility of the two components is large. This low tensile ductility can be related to the susceptibility of the less ductile layer to early cracking.

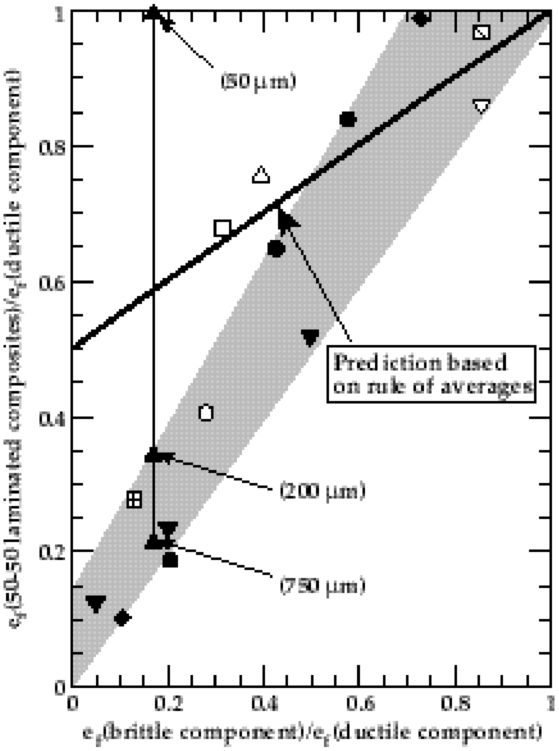


Figure 16. Experimental tensile elongation to fracture e_f of thick layer laminated composites containing 50 volume percent of each component, normalized with the elongation of the ductile component, compared with prediction based on rule of averages. Symbols same as in Figure 15. [1]

The most profound observation in Fig. 16, however, is seen for the data on the UHCS/brass laminate (solid triangles). The results show that the tensile ductility of the laminate can be either less or greater than the prediction from the rule of averages. These results are related to a layer thickness effect on the ductility of laminated composites. When the layer thickness is 750 μm, the tensile ductility is 13%; when the layer thickness is 200 μm, the tensile ductility increased to 21%; and when the layer thickness is 50 μm, the tensile ductility is 60%.[47]

This trend is attributed to the greater difficulty in delamination as the layer thickness is reduced. Interfacial delamination is suppressed with decreasing layer thickness because the residual stress (from thermal contraction differences of the components) is decreased.[47] Inhibition of delamination prevents neck formation in the UHCS layers which would otherwise create hydrostatic tension in the neck region in these layers leading to crack initiation and failure. The extension of uniform plastic flow in the strong UHCS layers, results in enhancing the tensile ductility of the laminate. Tensile ductility has also been studied in Ti alloy laminates containing alternate layers of an α−β Ti alloy (Ti-Al-Cr-Mo-V) and a medium strength β−Ti alloy (Ti-Al-Mn) containing different volume fractions of the component materials.[48] In general, at all volume fractions, the laminate showed increasing tensile ductility with decreasing layer thickness.

It is possible that this type of benefit would apply to historical artifacts, by clearly improved resistance to delamination through the mechanisms described above. Thus, some insights into the ductility of ancient laminated artifacts are possible.

Tensile Behavior at High Temperatures: The tensile behavior of metal laminated composites at elevated temperature has been principally studied for potential superplastic behavior. The studies have shown how a non-superplastic material can be made superplastic by lamination.[49-53] The basis for this success is the achievement of a high strain rate sensitivity laminate by the appropriate choice of components. High strain rate sensitivity, defined by the exponent, m $(d \ln \sigma / d \ln \dot{\varepsilon})$, inhibits neck formation and leads to high tensile ductility. Other laminate studies have involved tests to assess their stress state during compression deformation under testing perpendicular to the laminate direction.[54] Such tests are useful as a guide to optimize open die forging or rolling of laminates.

For example, it has been shown that a non-superplastic material (interstitial free iron) could be made superplastic by lamination with a superplastic material (fine grained UHCS). It was shown that the predicted behavior of the composite was readily determined by assuming isostrain deformation of a laminated composite containing two components. A schematic illustration of the laminated composite is given in Fig. 17(a). The deformation of such a composite can be described by a mechanical analogy consisting of two dashpots in parallel as shown in Fig. 17(b). Since the dashpots are forced to flow at the same rate, the dashpots are said to be interdependent. Thus, in this case, the stronger dashpot controls the flow rate of the two-dashpot model.

Fig. 17(c) illustrates the individual behavior of the two components, plotted as the logarithm of strain rate as a function of the logarithm of modulus compensated stress. The slope $(d \ln \dot{\varepsilon} / d \ln \sigma)$ is the stress exponent n, which is equal to the reciprocal of the strain rate sensitivity exponent m. As can be seen in Fig. 17(c), the superplastic material exhibits a low slope, that is, a low value of n, typically 2 (or, equivalently, a high value of m, typically 0.5). A low n is indicative of deformation by grain boundary sliding; and superplastic behavior, as demonstrated by high tensile elongation, is expected. The non-superplastic material exhibits a high slope (that is, a high stress exponent, typically n=8), which is indicative that deformation is by dislocation slip processes, and normal elongations are expected.

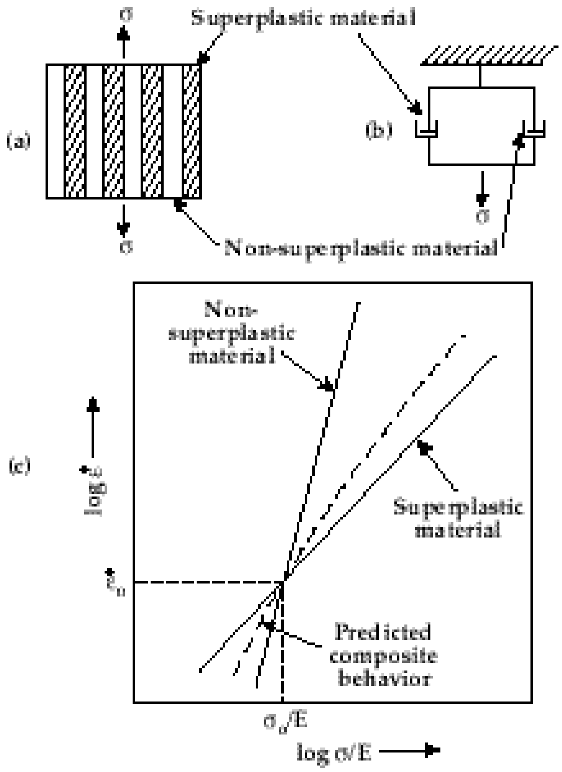


Figure 17. Schematic representations of (a) isostrain testing orientation of laminated composites; (b) mechanical analogy of deformation of two component laminated composite (in isostrain orientation, analogy consists of two dashpots arranged in parallel and subjected to a stress σ); and (c) predicted strain rate-stress behavior of each of the two components and the overall behavior of the laminated composite. Behavior is predicted to follow that of the stronger of the two components.[1]

The laminated composite, shown by the broken line in Fig. 17(c), follows the behavior of the stronger of the two components. The strength of the laminated composite is predicted from the law of averages, that is:

$$\sigma_c = f_{gbs}\, \sigma_{gbs} + f_{sl}\, \sigma_{sl} \qquad (2)$$

where σ_c is the strength of the composite, σ_{gbs} and σ_{sl} the strengths of the components controlled by grain boundary sliding and dislocation slip, respectively, and f_{gbs} and f_{sl} their volume fractions, respectively. The highest elongation achieved in the experimental interstitial free (IF) iron/UHCS composite system was 430% elongation at 650°C in contrast to 100% elongation for IF iron alone.

Thus, it is possible that, in the case of ancient laminated composites that contained an ultrahigh carbon steel component, superplastic forming was a component of the laminate forming. The monolithic superplastic behavior of modern UHCS containing a surface damask has been demonstrated.

Impact Behavior: While the understanding of the sources of fracture toughness in LMC systems is at a relatively early stage, there are some areas that are emerging as important.

One of these relates to the role of delamination between layers. Delamination appears to play an important role in activating key extrinsic toughening mechanisms, particularly the crack blunting, crack front convolution, and local plane stress mechanisms. The energy absorption due to delamination itself appears to be less critical than the role it has in the initiation of these extrinsic mechanisms. Certainly, available experimental data support the fact that increasing toughness is seen in laminated metal composite systems with greater amounts of delamination. In fact, increasing fracture toughness with increasing fraction of reinforced layers has been observed in one system,[55] with this seemingly counter intuitive increase being the result of increasing activation of the extrinsic toughening mechanisms enabled by greater extent of delamination in the samples containing a greater fraction of reinforced layers.

Notch Impact: One of the attractions of developing laminated composites is that they can exhibit a very high notch impact resistance. It is this area of property improvement that is perhaps not intuitive in its application to ancient welded laminated products.

In the area of notch impact resistance, high impact strengths have been obtained in laminated composites based on UHCS. A dramatic example is shown in Fig. 18 for a UHCS/mild steel laminate.[56] The results are for Charpy V-notch impact tests on a laminated composite of UHCS/mild steel, in the crack arrester orientation. Also shown in the figure are the results of Charpy V-notch impact tests on the monolithic UHCS and the monolithic mild steel. It is worth noting that each of these monolithic samples was thermo-mechanically processed in an identical manner to the corresponding individual steel layers in the laminated composite. Thus, the microstructure of the monolithic UHCS was identical to that within the individual layers of UHCS in the laminated composite; the same correspondence was true for the mild steel.

The Charpy V-notch impact properties for these materials, i.e. the 12-layer laminated composite of UHCS/mild steel, the monolithic UHCS, and the monolithic mild steel are very different. The laminated composite, in the crack arrester orientation, showed greatly improved impact resistance characteristics compared with either of the monolithic steels. For example, the upper shelf energy of the UHCS/mild steel laminated composite is ~325J compared with 190J for the monolithic mild steel and 75J for the monolithic UHCS. In addition, the 20J ductile-to-brittle transition temperature (DBTT) is –150°C for the laminated composite compared with –100°C for the monolithic mild steel and 0°C for the UHCS.

The dramatic improvement in the impact properties of the laminated composite is a result of notch blunting by extensive delaminations that occur on either side of the initial crack direction in all samples. The samples, however, remained intact and delamination was confined to the center regions adjacent to the initial crack direction. The ability to delaminate is a key factor in controlling the impact characteristics of the laminated composite.

The above hypothesis was tested by heat treating samples of the UHCS/mild steel laminated composite to above the A_1 temperature. This heat treatment was based on a previous observation[57] that the bonding between the UHCS layers in a UHCS/UHCS laminate could be improved by such a heat treatment cycle. This specific heat treatment cycle involved heating the UHCS laminate to just above the A_1 transformation temperature, e.g. 770°C, quenching in water, and then annealing at 650°C. This procedure does not affect the strength or microstructure of the bulk UHCS, but does improve the bond strength. This improvement in bond strength has been presumed to be related to the following factors: interdiffusion between individual lamellae, that occurs at temperatures above the A_1 temperature; the effect of transformation on the interface; and grain boundary migration across the interface. A similar heat treatment procedure was carried out on the UHCS/mild steel laminated composite in the study.

The results of Charpy V-notch impact tests on the UHCS/mild steel laminated composite, in the crack arrester orientation after heat treatment (strong interfaces), are shown in Fig. 19. Also shown are the results on the as-rolled samples (weak interfaces) taken from Fig. 18. The impact properties of the laminated composite are significantly degraded by heat treatment. Macrographs of these samples are shown in Fig. 20. The as-rolled sample tested at –79°C with an impact energy of >325J is shown in Fig. 20 (top). The fracture behavior of the as-rolled sample (weak interfaces) can be compared with the heat-treated sample (strong interfaces), shown in Fig. 20 (bottom), which has an impact energy of 18J at –79°C. It is clear that delamination is significantly reduced following heat treatment. It was concluded that the loss in impact strength following heat treatment is a result of improving the interface strength.

It is therefore implied in Figs. 19 and 20 that the excellent Charpy V-notch properties of the as-rolled laminated composite in the crack arrester orientation are a result of the effects arising from delamination (e.g. by crack blunting or increased energy absorption during delamination). If this is so, then the composition of the interleaf material in laminated composites containing UHCS should not have a great influence on their impact properties. One way to examine this proposal was to replace the mild steel layers in the UHCS/mild steel laminated composite with UHCS layers. The impact properties of such a UHCS laminate should be similar to the UHCS/mild steel laminated composite.

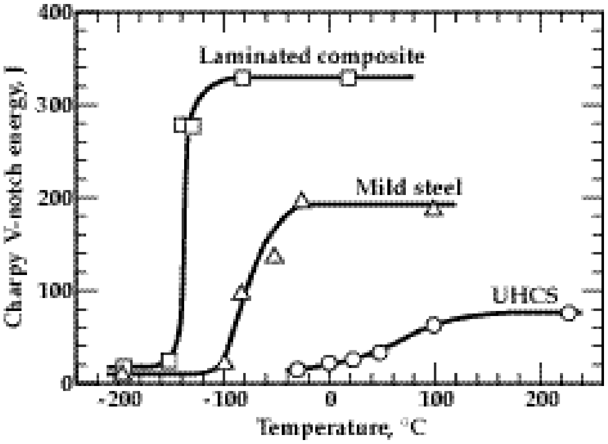


Figure 18. Charpy V-notch impact test results for 12-layer laminated composite of UHCS/mild steel in crack arrester orientation and for mild steel and UHCS monolithic steels.[56]

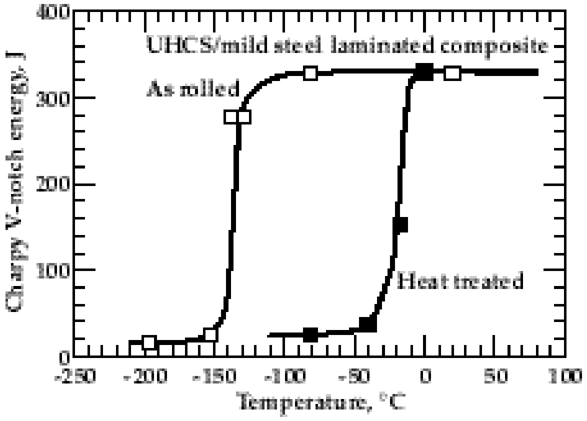


Figure 19. Charpy V-notch impact test in crack arrester orientation for 12-layer laminated UHCS/mild steel composites, both in as-rolled (weak interfaces) and heat-treated (strong interfaces) conditions. Degradation of impact properties occurs as result of heat treatment.[56]

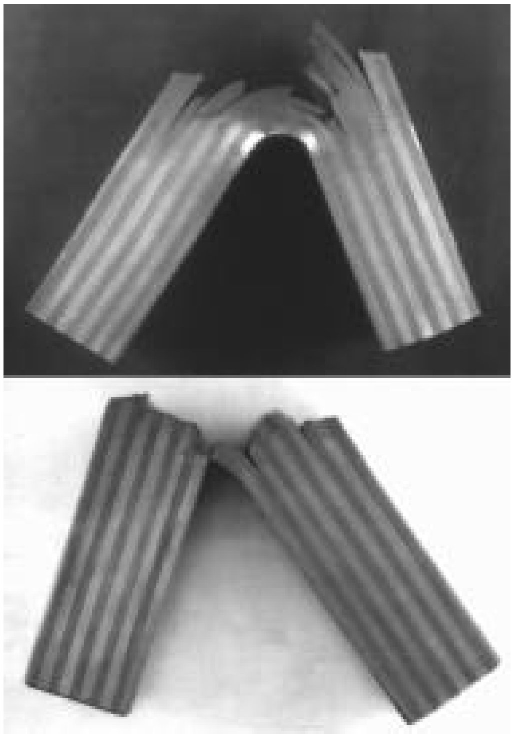


Figure 20. Macrographs comparing fracture behavior at –79°C for 12-layer laminated UHCS/mild steel composite. Extensive delamination occurs in (top) as-rolled (weak interfaces) condition, but is absent in (bottom) heat-treated (strong interfaces) condition. [56]

To test this hypothesis, an 8-layer laminate of UHCS was prepared. The transition temperature (~ –150°C) and the upper shelf energy (>325J) were found to be the same for the two laminated materials. The lower shelf energy, however, was greater for the UHCS/UHCS laminate (~80J) than for the UHCS/mild steel laminated composite (~14J). The results indicate that excellent impact characteristics are achieved in UHCS laminated composites irrespective of the interleaf material (i.e. mild steel or UHCS).

In recent years, the precise role of the interleaf materials — for example, Hadfield manganese steel (HMS)[58], nickel-silicon steels[59], and brass[60] — on the impact properties of UHCS has been studied in more detail. The notch impact curves for different interleaf material in the heat-treated condition have been studied. The UHCS/brass combination provides the best impact properties at low temperature. This is because brass does not exhibit a DBTT and thereby contributes to the blunting of propagating notches.

Recently, prototype steel laminated composites have been made essentially by joining galvanized steel sheets. In this manner, a laminated composite consisting of steel layers and zinc layers separated by zinc/steel interdiffusion layers is created. The impact resistance of these composites has also been shown to be enhanced by delamination under impact conditions.

Ballistic Impact: The response of metallic laminates to ballistic impact has been studied by several investigators[61-64] and, in general, the results have shown that laminate plates can be designed to increase the amount of energy absorbed during impact and thus improve the materials resistance to penetration, perforation, and spall relative to non-laminated targets.

Lamination can change the mechanism of failure during ballistic impact, which can dramatically increase the amount of energy absorbed. Typically, during ballistic impact by blunt projectiles, monolithic aluminum alloys and aluminum matrix composites fail by low energy failure mechanisms involving shear localization and the acceleration and compression of a shear plug. The result is a shear plug of material being ejected from the target with relatively low energy. However, in laminate form, materials which fail by shear localization during ballistic impact, can undergo local delaminations at component interfaces. These local delaminations reduce the stiffness of individual layers, which allow them to bend and increase the volume of material absorbing energy during impact. The result is that significantly more energy is required for penetration and perforation.

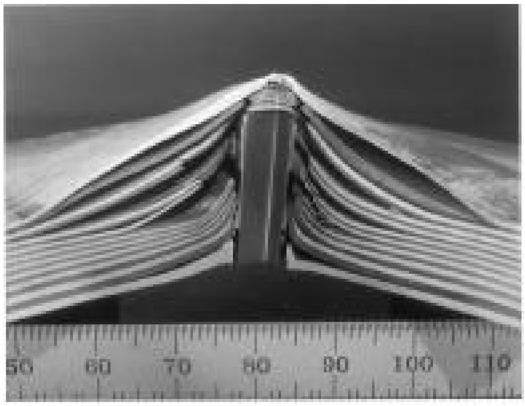


Figure 21. Laminated metal composite target impacted by hardened steel projectile travelling at 400 m s^{-1}. Dark layers are 6090–SiC(25p)–T6 and light layers are 5182; mm scale.[1]

An example of the ballistic performance of a LMC target is shown in Fig. 21. The LMC, which consists of alternating layers of 6090–SiC (25p)–T6 (dark layer in Fig. 21) and 5182 (light layers), has been impacted by a rigid hardened steel projectile travelling at 400 m s^{-1}. Homogeneous plates of either component material would be perforated by the rigid projectile at this velocity, with intense shear and little energy dissipated by the target. For the LMC target, the outer layers on the impact side show a shear intensive failure representative of perforation modes typically encountered in homogeneous targets. Layers deeper in the target, however, show increasing amounts of delamination and layer flexure, with the bottom layers undergoing significant biaxial stretching. These flexure stretching deformation modes dramatically increase the volume of material participating in the penetration process and the amount of energy required for perforation of the target.

Laminate target performance of materials containing different adhesively bonded layers, during ballistic impact has shown that toughness is a strong function of interfacial strength. Thus, numerous studies have illustrated the benefits to fracture resistance that can be achieved via the creation of a layered or laminated structure. Improvements to the fracture resistance have been measured using a variety of means including: tension testing, Charpy impact testing, ballistic testing, and variants of tests typically used to determine the fracture toughness and/or flaw tolerance of structural materials. Charpy impact and ballistic testing results have been reviewed above.

Toughening Mechanisms: Toughening in LMCs can arise from many different sources. Recent work has shown that toughening in materials can result from two different types of mechanisms — intrinsic and extrinsic.[65, 66] Intrinsic toughening results from the inherent resistance of the microstructure to crack growth and thus is influenced by such microstructural characteristics as grain size, particle spacing, particle size, etc. Extrinsic toughening, on the other hand, results from mechanisms that reduce the local stress intensity at the crack tip and thus the local “driving force” for crack growth. The distinct layers present in LMCs toughen these materials by various extrinsic mechanisms, which are shown schematically in Fig. 22. The laminate orientation that will be influenced by the mechanism, the sensitivity of the mechanism to the volume fraction of the component materials, and the mechanisms that can produce R-curve behavior are indicated in the figure. Numerous other extrinsic and intrinsic toughening mechanisms have been identified in composites and monolithic materials[65, 66], and these can provide additional sources of toughening in LMCs.

Crack deflection: In many laminate systems, layer delamination can occur ahead of an advancing crack or as the result of a crack encountering an interface. These local delaminations can result in crack deflection which can significantly reduce the mode I component of the local stress intensity because of the large deviations in crack path (up to 90° in the crack arrester orientation) that are possible. These crack path deviations cause the crack to move away from the plane experiencing maximum stress.

Crack blunting: In a LMC, crack blunting can result when a propagating crack encounters a ruptured region. When the advancing crack encounters the ductile layer, the crack is deflected (due to the delamination) and blunted (due to the ruptured layer). Further crack growth requires re-nucleation of the crack in the MMC layer. This arresting and re-nucleation process results in a significant increase in the amount of energy required for crack growth.

Crack bridging: In this mechanism, unbroken individual layers span the wake of a crack. Growth of the crack requires stretching of these bridging ligaments. It is important to recognize that for crack bridging to occur, the bridging ligaments must have sufficient ductility to avoid fracture at or ahead of the advancing crack tip. Thus crack bridging, as opposed to crack blunting, will occur when ductility or toughness differences exist between the component layers.

Stress redistribution: Delamination can also provide extrinsic toughening by reducing the stresses in the layers ahead of the advancing crack. This mechanism, referred to as stress redistribution in Fig. 22, has been recently studied both theoretically and experimentally for metal/ceramic laminates. In these studies, delamination was found to be significantly more effective than slip in reducing the stress ahead of the crack.

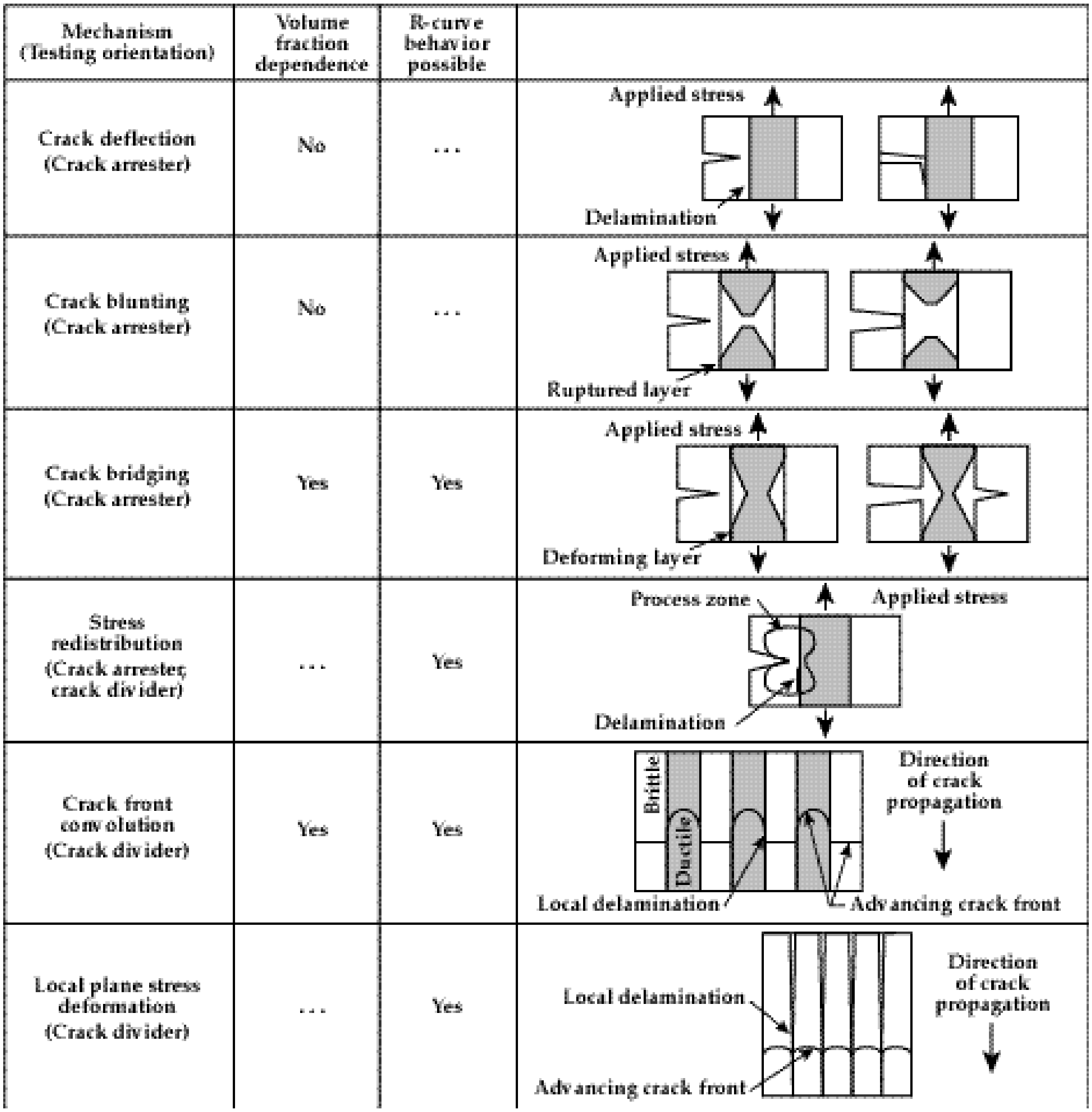
Crack front convolution: This mechanism is unique to LMCs containing layers with dissimilar ductilities that are tested in the crack divider orientation. In this mechanism, the crack front in the less ductile component of the laminate leads the crack front in the more ductile component. The shape of the crack front is highly convoluted with the depth of the convolutions related to the extent of delamination at the interfaces. Overall crack front growth is retarded by the plastic tearing required for crack growth in the more ductile layer.

Local plane stress deformation: For laminates tested in the crack divider orientation, if substantial delamination occurs at the crack tip, then layers can deform individually under plane stress rather than plane strain conditions. This causes individual layers to fail in shear (as opposed to a flat fracture) and can substantially increase the stress required for crack growth. Thus, toughness measured on thick samples was equal to the high toughness that would have been obtained for the individual thin layers under plane stress conditions.

Summary — Fracture Resistance: The work thus far on toughening in LMC systems clearly indicates that different mechanisms are dominant depending on the orientation of the crack relative to the layer architecture. In the crack arrester orientation, it appears that crack blunting and deflection are the dominant mechanisms, and can produce appreciable increases in toughness.

In the crack divider orientation, increases in fracture toughness relative to unlaminated systems have tended to be more modest. The dominant mechanisms in this case are crack front convolution and local plane stress deformation. Further improvements in the toughness in the crack divider orientation are desirable as this is often the key orientation for many structural applications.

It is difficult to generalize about the benefits of lamination with respect to fracture resistance in ancient artifacts. Clearly, some of the above mechanisms could well apply. However, it is probably necessary to evaluate each ancient material for such fracture resistance improvements. The Japanese sword for example is a laminate at several levels.



Mechanism (Testing orientation)	Volume fraction dependence	R-curve behavior possible
Crack deflection (Crack arrester)	No	. . .
Crack blunting (Crack arrester)	No	. . .
Crack bridging (Crack arrester)	Yes	Yes
Stress redistribution (Crack arrester, crack divider)	. . .	Yes
Crack front convolution (Crack divider)	Yes	Yes
Local plane stress deformation (Crack divider)	. . .	Yes

Figure 22. Toughening mechanisms for laminated metal composites.[1]

Damping Properties: In general, monolithic materials that have good structural properties (such as steel and aluminum) have poor damping behavior. On the other hand, materials with poor structural properties (such as lead and plastic) generally have high damping capacity. Laminated metal composites have the potential to improve the damping response of these structural materials through the activation of additional damping mechanisms. These vibration suppressing mechanisms are additive to the ones active in the individual layers from which the LMC is composed. The mechanisms resulting from lamination are associated with the planar interfaces and abrupt changes in elastic constants on going from one layer to another. Typically planar interfaces in materials have been high sources of damping in materials. Examples include the motion of magnetic domains in a stress field, the sliding of grain boundaries subjected to a shear stress, and the flow of thermal currents across heterogeneous regions.

Recent damping studies on a UHCS/brass system have shown that LMCs have the potential for excellent structural properties and high damping capacity. In this study, damping measurements were made in two widely different frequency regimes — low frequency (2-40 Hz) and ultrasonic frequency (2.25 MHz). In both cases, the laminate had higher damping capacity than the component materials.

In at least one case — the Japanese sword — claims of improved damping have been made for a blade consisting of an iron core in a high carbon steel sheath.

Final Comments

A summary is given in Table 3 of modern laminated composites. There are many reasons to laminate materials in both ancient and modern materials. An assessment is given in Table 4 of the possible motivations or benefits of lamination. As can be observed, the motives for lamination vary considerably and depend very much on the material under consideration.

Acknowledgement

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Table 3. Some Examples of Modern Laminated Composites

Material	Approximate Era	Composition (where known)	
		Layer A	Layer B
UHCS Mild Steel	1979 - Present	1%C	AISI 1020, 0.2%C
UHCS Interstitial Free Iron	1984	UHCS	~0%C
UHCS HMS	1990	UHCS	Hadfield manganese steel
UHCS Ni/Si	1992	UHCS	Ni – Si
UHCS Brass	1992	UHCS	Al – bronze, brass 70%Cu – 30%Zi
UHCS 304 SS	1997	UHCS	304 stainless steel
UHCS Fe – 3 Si		UHCS	Fe – 3%Si
Former Soviet Union Oil Pipes	Present	oil pipe steel	same composition
Former Soviet Union Explosive Forming	Present	tool steel/tool steel	Cu/Al
Moscow Steel and Alloy Institute Concentric Tubes	Present	2.1 – 2.6%C	0.6%C
Modern Japanese Sword	Present	see ancient Japanese Sword	
Norwegian 3-layer Blades and Japanese Chisels	Present	A — B — A laminate type A — low carbon or stainless B — high carbon tool steel	
Modern Damascus Steel Pattern Welded Knives	1970-Present	see Table 4 in accompanying paper by Wadsworth and Lesuer	

Table 4. Possible Motivations for Laminated Materials

Laminated Artifact	Limited Material	Processing to Make Bulk Material	Tensile Strength	Improved Toughness	Improved Damping	Attractiveness, Quality
Gizeh Iron Plate	✓					
Achilles' Shield				✓		?
Adze	✓	✓		?		
Chinese Refinings	?	✓	?	?		
Merovingian	✓	✓	?			✓
Japanese Sword			✓	✓	✓	✓
Thailand Tools	✓	✓	✓	?		
Indonesian Kris	✓	✓	?	?		✓
Halberd	✓			✓		
European Gun Barrels			✓	✓		✓
Shear/Double-Shear Steel		✓	?	✓		
Chinese Pattern Welded			?	?		✓
Persian Dagger				?		✓
FSU Materials			✓	✓		
Modern Knives						✓
Modern Chisels				✓		

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