A NEW 925 SILVER ALLOY WITH INCREASED TARNISH RESISTANCE : FROM R&D OVER REAL-LIFE TESTS TO MANUFACTURING

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ABSTRACT

A new silver alloy has been developed with increased long-term tarnish resistance comparable to low carat gold alloys and which requires no additional protective coatings. The paper reports on the alloy development, balancing hardness, manufacturing properties, tarnish resistance as well as metal costs. Results of accelerated lab tarnish tests are presented and compared with extensive real life, long-term wear tests. Finally examples of manufacturing trials from cooperating jewelry and watch manufacturers are presented. The outcomes are discussed keeping in mind consumer expectations and market approach.

INTRODUCTION

Although substantial progress has already been made in understanding silver tarnish phenomena and in development of alloys with increased tarnish resistance, the vast majority of silver jewellery produced (~ 7000 tons/year worldwide) still is Ag- or Rh-plated and coated with protective layers. Alloys with true, long-term tarnish resistance should not require any such protective coatings, just like most medium- to high-karat gold alloys. There is a long-standing market quest for “natural” silver jewellery, without such surface coatings and with the original, unique sterling silver colour. However, even if such an alloy would exist, how much extra cost a consumer is willing to spend if that alloy contains expensive additions ? And how can the alloy supplier or jewellery manufacturer proof long-term tarnish resistance when no accelerated lab test seems to exist that reliably simulates real-life behavior? Apart from an already long list of desired alloy properties, such questions have to be kept in mind during alloy development.

PREVIOUS WORK

Probably all the potential aspects around tarnishing of silver alloys have already been the focus of in depth R&D and numerous corresponding presentations at the Santa Fe Symposium (1-14). Based on the published work combined with a look back to now ~ 25 years of practical, real-life experience with so-called tarnish- or oxidation-resistant alloys, the present state-of-the-alloy-art can be briefly summarized as follows:

- tarnishing of 925 Ag alloys can be delayed but not completely avoided
- commercially available, so-called “tarnish-resistant alloys” should instead and more correctly be referred to as alloys with “increased tarnish resistance”;
- despite of in part excellent results in a particular accelerated lab test, numerous so-called tarnish-resistant alloys do not perform significantly better or even less good than a standard sterling silver alloy (binary 925 Ag-Cu) in another lab test at different

conditions, during long term tests in real environments or simply by experience in real life;

- the metallurgical state of an alloy or jewelry item affects tarnish properties: as-cast items usually possess lowest tarnish resistance due to the potential presence of porosity or local compositional inhomogeneities; solution-annealed, hence homogenized material usually performs best, while cold-working or treatments like age-hardening may decrease tarnish resistance due to re-introduction of inhomogeneities.

Also about tarnish testing we can draw some basic conclusions:

- there is a general lack of agreement and standardization about tarnish test procedures (incl. sample preparation) within the jewellery industry, despite of several attempts to address this and despite of the general availability of numerous existing norms and guidelines;
- a single accelerated tarnish test that reliably predicts long term tarnish resistance of an alloy or a jewellery item in real environments does not exist;
- a set of multiple different accelerated tarnish tests addressing different tarnish mechanisms (formation of sulfides, chlorides, nitrides and oxides) is required to assess tarnish resistance most reliably at least on lab scale;
- long term tarnish tests in real, natural and industrial environments suffer from a lack in repeatability and statistics, but can give useful and complementing insight; for an alloy or jewelry supplier the final judgement basically is only possible based on long-term customer feedback.

Back to alloy properties, the “design” of silver alloys with increased tarnish resistance is based on one or more of the following principles (6, 9, 10, 11, 14):

- addition of elements that react with atmospheres, especially oxygen, and form colorless, protective passivating coatings
- minimization or better complete avoidance of dual-phase structures, like those usually present in a binary 925 Ag-Cu alloy ,
- precious metal additions with higher nobility than silver
- reduction of impurities and refinement of grain size to minimize structural and compositional inhomogeneities.

Argentium ® alloys, that are often referred to as a benchmark for a highly tarnish resistant alloys, are based on the ternary Ag-Cu-Ge system. Their increased tarnish resistance mainly relies on the spontaneous formation of passivating Germanium-Oxide layers on the surface of a jewelry item, in combination with control of impurities and grain size.
For some years, Legor was actively involved in the further improvement of Argentium ® alloys. Starting from an original 925 alloy for universal application, an alternative alloy with slightly revised composition and improved investment casting properties was suggested. Also formulas with further increased tarnish resistance were introduced. These improvements relied on the reduction of the typical dual-phase structure by partially replacing Cu by Ag. Results for the corresponding 960 Argentium ® alloys were shown in a former SFS paper (9).

Beneficial effects of precious metal additions on tarnish resistance and hardening properties of silver alloys amongst others have been reported in (11). While high additions of e.g. 2,5-3 wt% of Pd provide exceptional hardness and hardening properties, the alloy costs would probably raise more than acceptable. Furthermore, according to own investigations, that high Palladium additions do not necessarily provide optimum tarnish resistance in different (test) environments (14).

THE PROJECT

Based on a thorough analysis of the sketched previous work, a multiple years project has been started in 2014 which consisted in the following main steps:

1. **R&D** on alloy formulations and suitable analytical lab methodologies (06/2014 - 06/2015)
2. **Real-life wear tests** (06/2015 - 03/2016)
3. **Manufacturing trials** with partner companies (06/2015 - 12/2016) incl. ongoing real-life wear or accelerated lab tarnish tests on manufactured parts
4. **Market launch** (2017)

The overall goal was to develop a sterling silver alloy with increased tarnish resistance at least comparable to a premium 9k white gold alloy which by experience does not require protective coatings and has seen longterm acceptance in the market. Furthermore hardness and general manufacturing properties should be comparable to a standard 925 Sterling silver. Due to the indicated restrictions of both accelerated lab tarnish tests as well as real-life wear tests, it was decided to run all test in a comparative way, i.e. in comparison to well-defined reference alloys listed in ***Figure 1.*** As a so-called "positive" reference, the formerly introduced 960 Argentium alloy was chosen, which can possibly be considered the most tarnish resistant alloy available at that time.

Figure 1 *Reference alloys, compositions in wt%*

	Ag	Au	Cu	other	
925 Ag **Standard Sterling Silver**	92,5	-	7,5	-	**Negative reference**
960 Ag-Cu-Ge Argentium® 960ArgPrem	96	-	2,5	1,5 Ge, Zn …	**Positive reference**
9kt premium white gold Legor WA12B1	53	37,5	2,5	7 a.o. Zn	**Benchmark**

EXPERIMENTAL METHODS R&D PHASE

In the R&D phase, samples of varying alloy compositions were prepared

- starting from raw materials and master alloys of common commercial purity
- by closed chamber induction melting in graphite crucibles
- by ingot casting of plate shaped samples, subsequently prepared by standard metallographic techniques
- later on also ring shaped jewellery samples by investment casting, subsequently surface finished by standard jeweller techniques.

In terms of alloy compositions, the R&D phase focussed on alloys <u>without</u> known additions like Germanium or Silicon, which would naturally form passivating surface oxide layers. Instead the partial up to complete replacement of Copper by additions of e.g. Tin, Zinc, Indium as well as combinations of such alloys with small additions of Palladium were studied.

Up to 6 different accelerated lab tarnish tests were carried out in order to investigate the tarnish resistance at different conditions, see the list in ***Figure 2.*** Routinely the first two listed tests were carried out for any new alloy compositions while selected alloys, then cast in ring shape, were submitted to the full set of tests.

Figure 2 *List of accelerated lab tarnish tests*

Type of Test	Standard Ref.	Time [h]
H_2S-Fume	**ISO 4538:1998**	**24**
Artificial Sweat Fume	**~ ISO 12870:2009**	**24**
UV	Internal Procedure	24
Artificial Sweat Contact	NFS 80-772:2010	24
Salt Spray Test (NSS)	ISO 9227:2012	96
Damp Heat with Leather	ISO 4611:2011	96

The first test on the list possibly is the most known one, where the sample surfaces are exposed to H_2S-vapour obtained by reaction of thioacetamide powder with a water solution saturated with sodium acetate at 75% humidity. This very strong test has been carried out up to 24 h, but results routinely are already checked after shorter duration.
During the 2nd test, also referred to "perspiration test", samples are exposed in a comparable way to an artificial sweat vapour, hence the presence of chlorides. This test derives from an ISO test standard originally developed for the spectacles industry, but different to the norm the samples are exposed to a saturated atmosphere only, while the original norm foresees direct contact with cloth soaked with the artificial sweat. This results in a less strong but more uniform tarnish attack.
An accelerated artificial sweat test where the samples actually are in direct contact with a soaked cloth is also carried out but according to a French norm, which is more widespread in Europe, and which originally has been developed for (gold-plated) watch cases.
The "salt spray test" is a well-known test for all types of metal surfaces but especially coatings, and in those cases is considered very relevant to reveal coating defects leading to pitting corrosion. For (un-coated) silver alloys it is relevant because it is particularly sensitive to (Cu-rich) second phases like in standard sterling or Argentium silver alloys.
A useful addition to the test portfolio is the exposure to damp heat in combination with e.g. leather. This particular test, originally developed and standardised for plastic material,

simulates strong humidity conditions in combination with the presences of substances which may release from materials like leather, textiles or packaging material.
Finally, the UV test is the only one from the list, which does not follow an existing suitable standard. It simulates the superimposed influence of heating up the surface and formation of ozone by UV exposure.

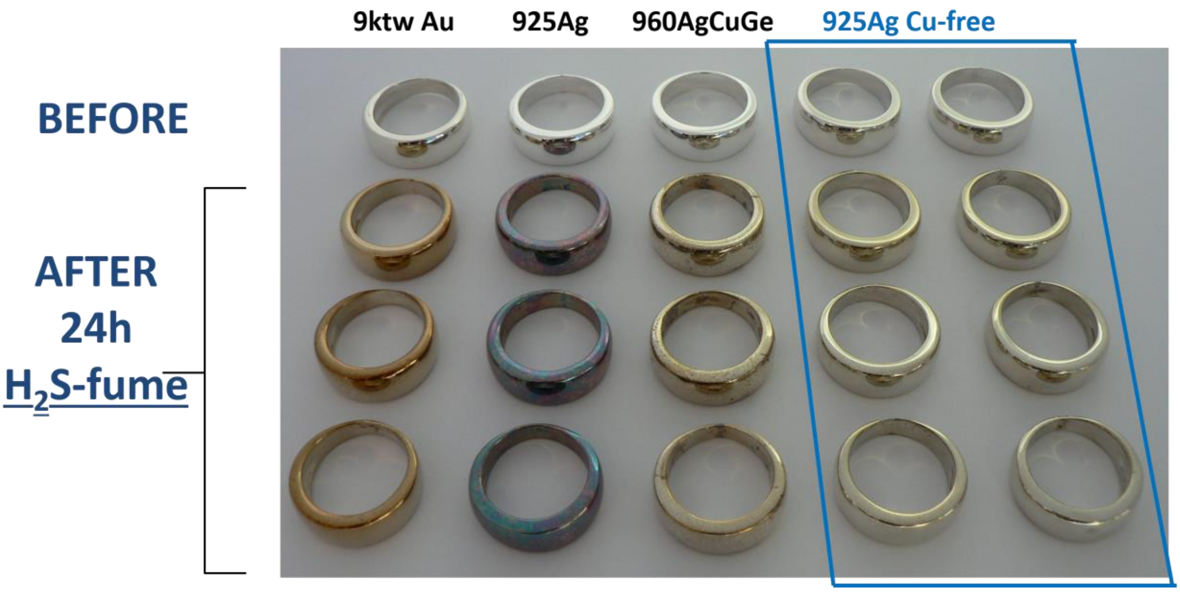
The samples surfaces were compared in a qualitative way always by visual inspection and photo documentation, while for selected test series quantitative results were collected via standardized conventional measurement of colour, more precisely colour variation.

RESULTS FROM R&D PHASE

While numerous results from the R&D phase have already been presented elsewhere (14), the present article focusses on the main outcomes.

During the accelerated lab tests, it was consistently seen, that 925 silver alloys which are completely free of Copper, show a comparably high tarnish resistance. In the H_2S-test, such alloys do not only outperform standard sterling or Argentium silver, but also the premium 9kt white gold alloy chosen as benchmark alloy (***Figure 3***).

Figure 3 *Results from H_2S-test*



More importantly, however, comparably high resistance and a performance matching the 9kt benchmark alloy is also observed during the artificial sweat tests, where alloys relying on passivating surface oxide layers are known to perform comparably weak (***Figures 4*** and ***5***).

The results obtained were considered a very strong experimental proof that second phases (Cu-rich) that usually are present in as-cast standard sterling silver as well as Argentium-type alloys, highly restrict tarnish resistance in some accelerated lab tarnish tests.

Figure 4 Results from artificial sweat fume -test

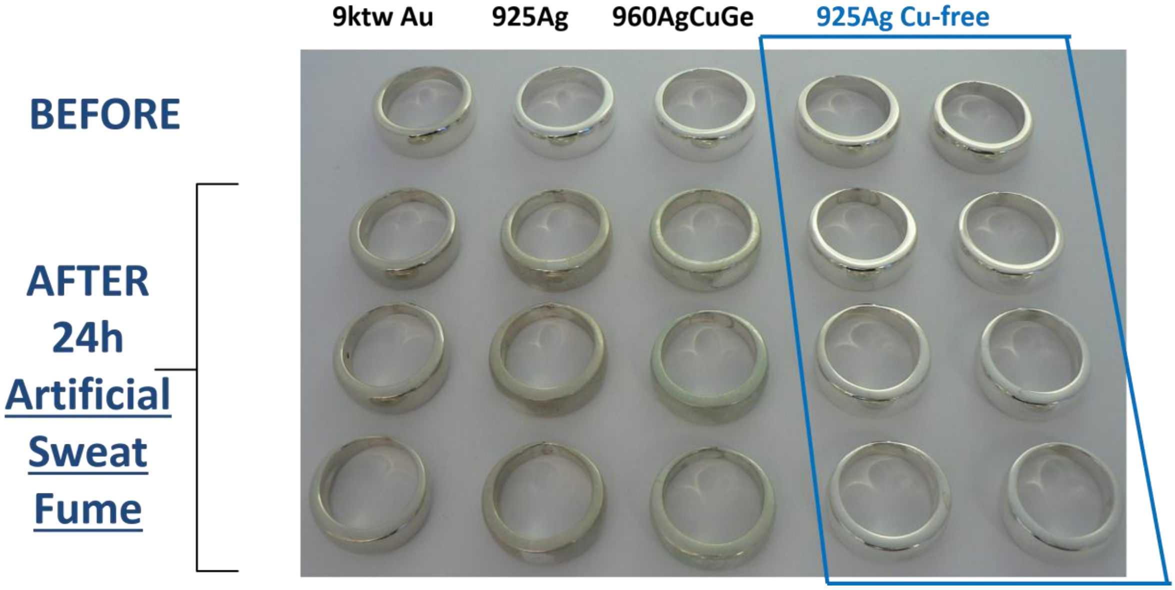
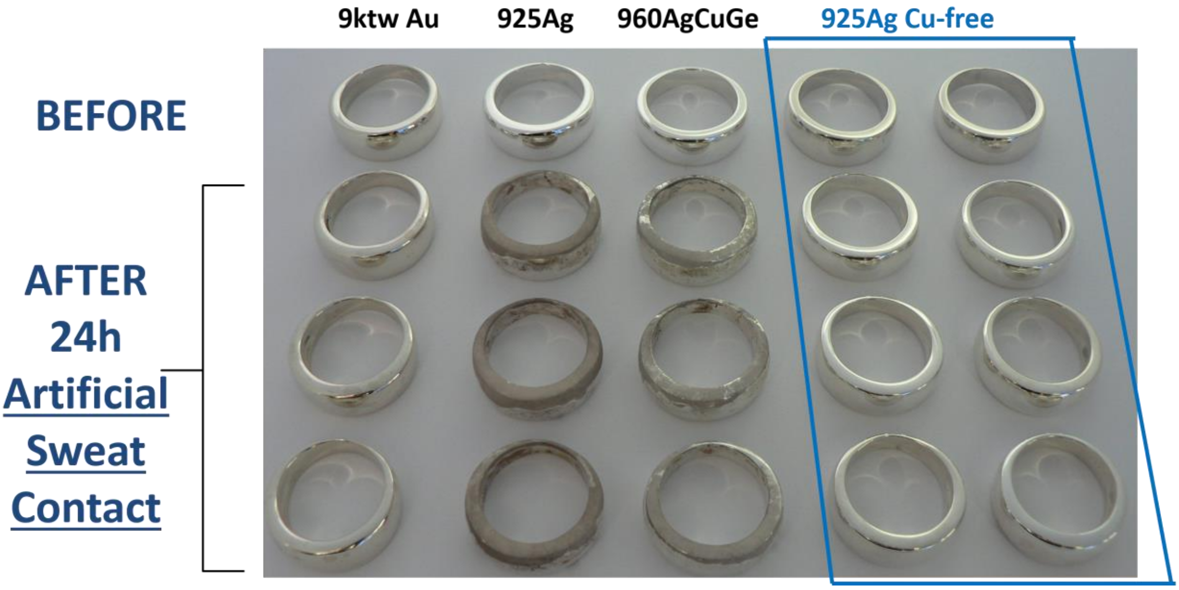
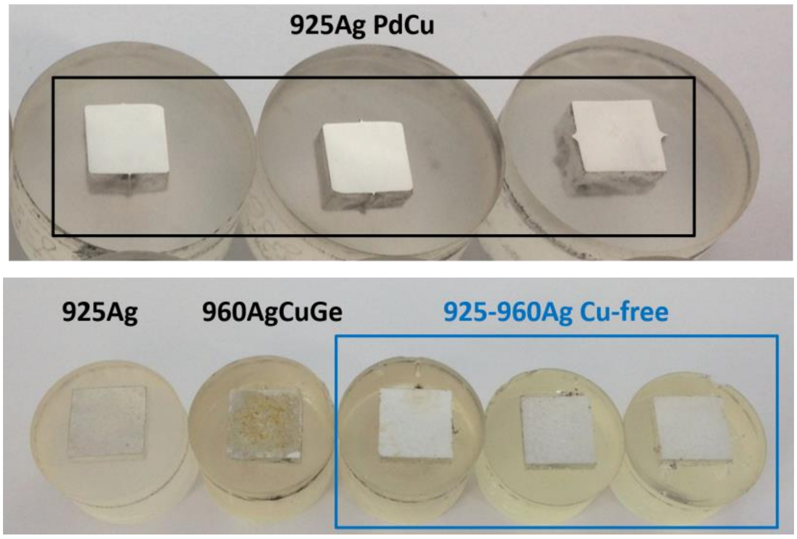


Figure 5 Results from artificial sweat contact -test



The already known downside of Cu-free 925 silver alloys is a low as-cast hardness, only slightly above that of pure silver namely in the range of ~ 40 HV. From previous work (e.g. 11) it was already known, that Pd additions can be useful for strengthening of 925 alloys where Copper is partially replaced by e.g. Tin. The own R&D revealed, that small Pd additions up to maximum 2 wt% are highly efficient in obtaining potential for significant age-hardening even in 925 alloys where Copper is completely replaced by additions of Tin, Indium, Zinc or alike, presumably by formation of uniformly distributed, nanometer sized intermetallic Pd-X (X=Sn, In, Zn) precipitates. Furthermore, similarly small additions of Pd proved to enhance tarnish resistance especially in artificial sweat tests and even if Copper was still present to some extent (***Figure 6***).

__Figure 6__ Results from artificial sweat fume -test, 24 h, influence of Pd additions



After actually 186 different alloy formulations tested the alloy composition range shown in ***Figure 7*** was defined, which based on the authors results provides an optimum combination of significantly increased tarnish resistance and mechanical properties. According to this, small remaining amounts of Copper may still be tolerated in the alloy, leading to a compromise of slight increases in as-cast hardness but also a slight deterioration of tarnish resistance if compared to alloys completely free of Copper. Several alloys from that range were chosen for the subsequent real-life wear tests.

__Figure 7__ Range of new 925 Ag alloy compositions (in wt%) with significantly increased tarnish resistance

	Ag	Pd	Cu	In, Sn, Zn, …
925 Ag New	92,5	0,5 - 2,5	0 - 2	3 - 7

The following ***Figures 8*** and ***9*** report further properties of such selected new 925 Ag compositions in comparison to a standard 925 sterling silver alloy. Notable are the bright silvery-white colour, the age-hardening properties from an as-cast state (quenched investment cast state, without solution annealing) as well as a comparably narrow width of the melting range. Furthermore, the tensile properties require particular attention, since both the yield strength as well as the tensile strength are comparably high, while the ductility (tensile elongation) remains high, too. The tensile properties were obtained on wires obtained after wire-drawing and a final soft-annealing thermal treatment typical for standard sterling silver. The data therefore indicate work hardening and soft-annealing behaviour of such new 925 Ag alloys which is markedly different to standard sterling silver.

***Figure 8** Thermal /colour properties of 925 Ag New compared to standard 925 sterling Ag*

	925 Ag Standard	925 Ag NEW
CieLab coordinates	L: 92,5 a: -0,1 b: 5,6 c: 5,6	L: 97 a: -0,3 b: 4 c: 4
Yellowness index	**YI: 11,5**	**YI: 7**
Liquidus Temperature °C	900	910-920
Solidus Temperature °C	780	850-870
Width dT (K) of melting range	**120**	**40-60**

***Figure 9** Mechanical properties of 925 Ag New alloys compared to standard 925 sterling Ag*

	925 Ag Standard	925 Ag NEW
Hardness as-cast HV	60 – 70	**50 – 60**
Age-hardening * HV	110 – 120	**110 – 130**
Yield Strength N/mm^2	120 – 135	**200 – 250**
Tensile Strength N/mm^2	250 – 275	**340 – 460**
Tensile Elongation %	25 – 30	**25 – 30**

*** directly from as-cast state => 1-step age-hardening treatment**

SAMPLES AND PROCEDURES : REAL-LIFE TEST PHASE

While the outlooks for a viable new 925 alloy with significantly increased tarnish resistance appeared good based on the R&D phase, an extensive Real-Life test phase was added, in order to check the evolution of tarnish during everyday use of such alloys.

Bracelets with beads made of new 925 Ag alloy compositions as well as reference samples of the formerly introduced reference and benchmark alloys (plus a 9kt yellow gold) were prepared **(Figure 10)**. Furthermore also samples of Cu-free alloys without Pd-additions were added to the bracelets. The beads were obtained by investment casting, an age-hardening treatment (where applicable) and standard jewellery surface finishing. No plating deposits or other anti-tarnish treatments were applied. Note that the bead design contains deep groves, which will later on highlight darkening due to tarnishing without wearing-off of such tarnish layers during everyday use.

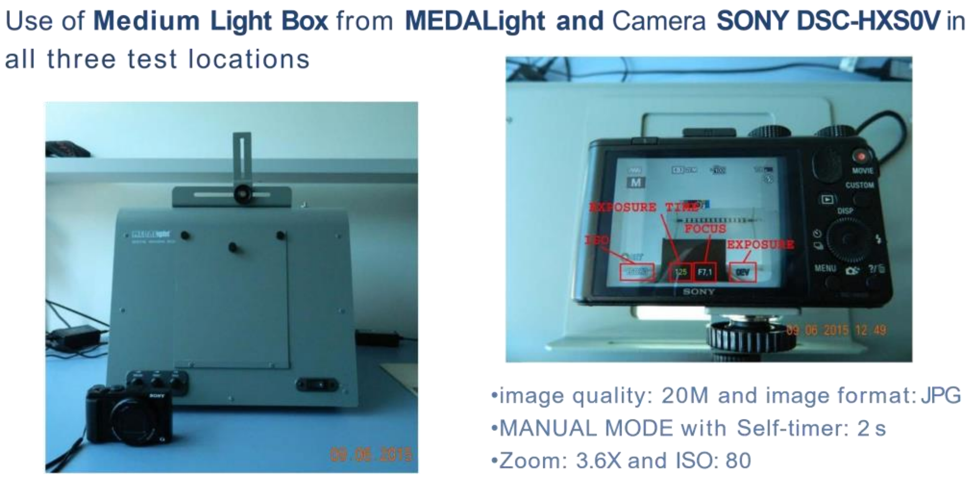
Bracelets were submitted to co-workers or their partners in areas with different climate and pollution conditions, as well different lifestyle:

2 bracelets >>> Bressanvido (Italy, Legor Group headquarter)
2 bracelets >>> Bangkok (Thailand)
2 bracelets >>> Mumbai (India).

In order to allow for reliable validation of the real-life results, a schedule with continuous (weekly) monitoring of colour variations with photo shootings under standardised conditions was agreed upon with the partners on-site in Thailand and India. This included supply of the same light box and camera as well as definition of camera settings for photo documentation (***Figure 11***).

Figure 10 *Bracelet as-prepared for real-life wear testing*

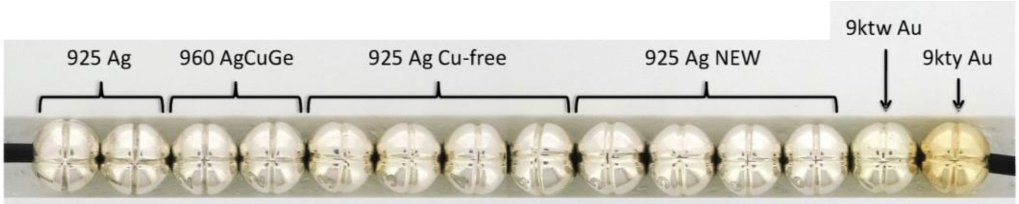
Figure 11 *Procedure for documentation of real-life tests*



RESULTS OF REAL-LIFE TEST PHASE

As expected, the results from the real-life tests differed significantly, clearly reflecting differences in lifestyle and area of the world, but also differences in alloy composition. Starting with Italy (***Figures 12*** and ***13***), after 2 weeks of use one bracelet still appeared almost new. The other one, however, which was worn during a 3-hours bicycle trip, showed considerable tarnishing for the 925 Ag reference alloys, while the new alloys remained as stable as the 9kt reference alloys, which also showed slight discoloration. After 9 weeks of use, the 1st (upper) bracelet now also revealed significant tarnishing for the 925 Ag reference alloys (darkening of the grooves), while the 2nd (lower) bracelet showed even more tarnishing, thanks to multiple visits to thermal baths. On the latter bracelet tarnishing is strongest for the 925 Ag reference alloys but present also for the '925 Ag New' as well as gold-based 9kt benchmark alloys. It is worth noting that the Cu-free alloys, without Pd-addition, perform worse than the '925 Ag New' alloys, of which one alloy stands out positively thanks to being completely Cu-free while containing also some Pd.

Figure 12: *Results of real-life test: 2 weeks Italy*



- Slight loss of brightness due to deterioration and samples abrasion

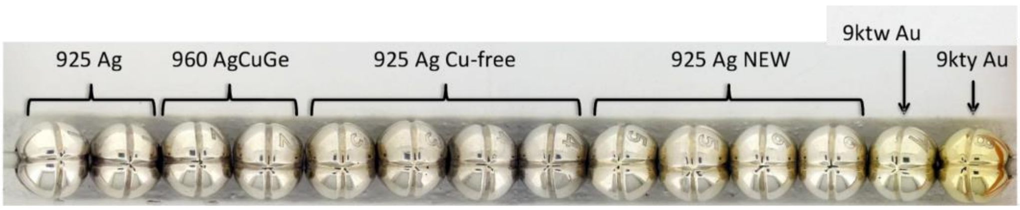
- Combined effect of: perspiration, temperature and UV radiation (worn during a three hour bicycle trip)

Figure 13: *Results of real-life test: 9 weeks Italy*

Loss of brightness due to abrasion; darkening of the "grooves".

Exposure to a sulfur-rich atmosphere (thermal baths)

Figure 14: *Results of real-life test: 9 weeks Thailand*



Loss of brightness due to abrasion; darkening of the "grooves "

Stronger darkening due to the daily use of chemical products used for cleaning in working environments;

Continuing with an example for Thailand, ***Figure 14***, a similarly strong influence of lifestyle can be observed, with the lower bracelet being worn by a person regularly handling cleaning chemicals during everyday business. Nevertheless, it can be stated also for these Asian real-life conditions, that the '925 Ag New' alloys perform better than the 925 Ag reference alloys and comparable to the 9kt gold benchmark alloys.

Finally, ***Figures 15*** and ***16***, compile pictures obtained for the same bracelets over a longer period for test persons in Italy and India, respectively. Clearly, the Indian real-life conditions led to the most pronounced tarnish reactions for all alloys, but still with '925 Ag New' alloys performing comparable to 9 kt gold benchmark alloys.

Figure 15: *Results of real-life test: up to 16 weeks Italy*

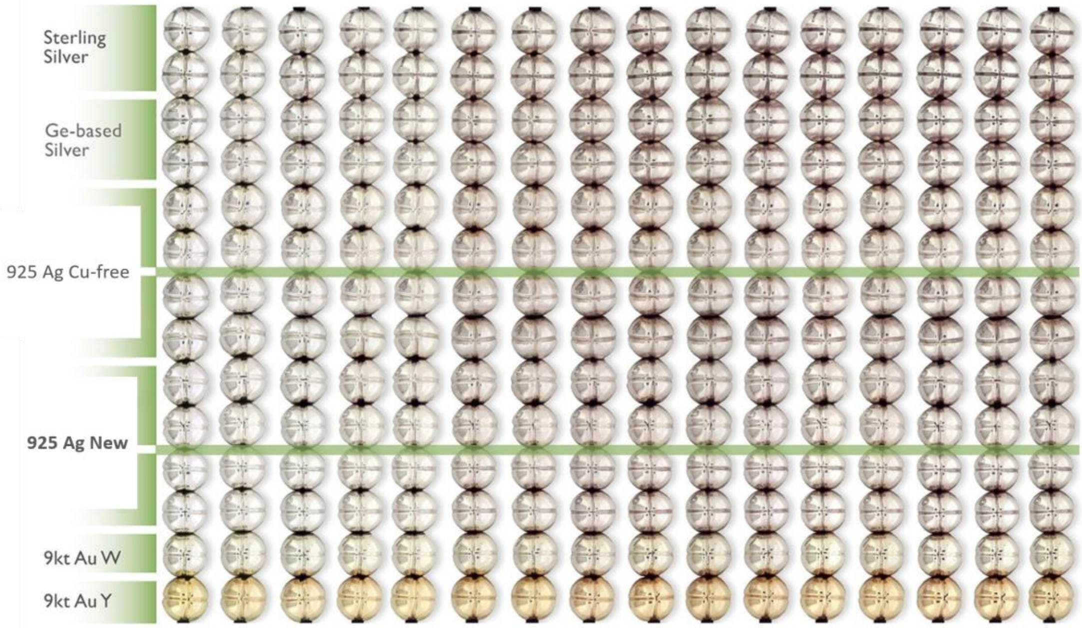
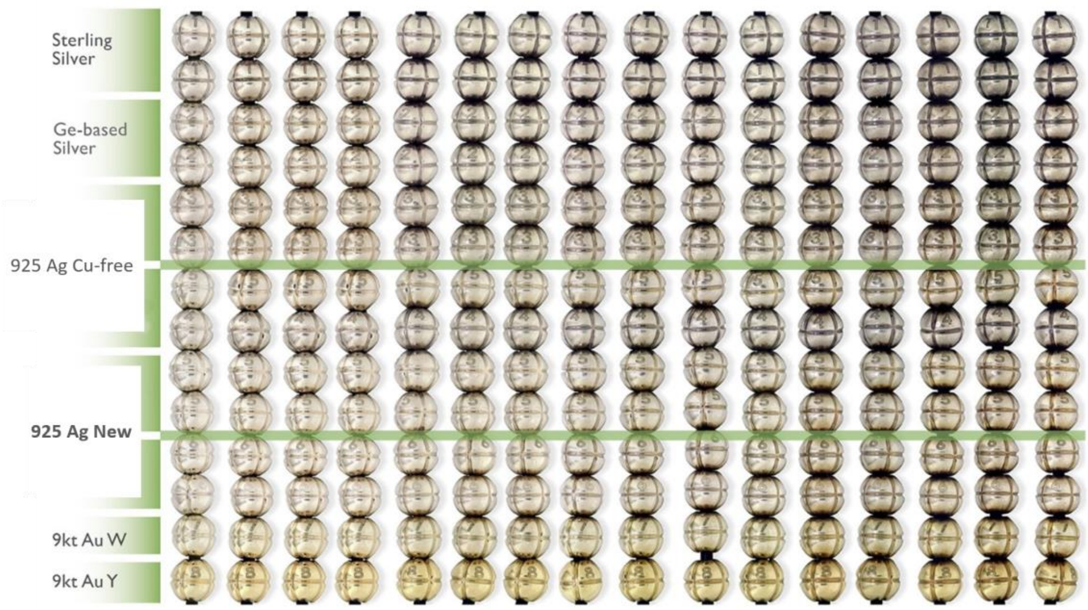


Figure 16: *Results of real-life test: up to 16 weeks India*



All-in-all, the results from the real-life tests can be considered consistent with those from the accelerated lab tarnish tests. This is good news for those not considering necessary to run real-life tests because they are labour-intensive and time-consuming. However, the authors believe that such tests are a relevant and useful extension of the pure R&D and lab work. On the one hand they increase the trust in the properties of a newly developed alloy. But they also provide a guideline for realistic expectations on an alloy with increased tarnish resistance, which as-stated already in the beginning, simply can not be considered completely tarnish-resistant in all possible real-life conditions.

MANUFACTURING TRIALS BY PARTNER COMPANIES

Eventually, and before market launch, regular sized production batches of selected ‘925 Ag New’ alloys were prepared and provided to cooperating companies interested in testing the newly developed alloys with view to overall casting and working properties in regular manufacturing environments. Samples from such trials in part were also submitted to accelerated lab tarnish tests as well as some real-life wear test, which were not documented with the former systematics, however.

Rings and bracelet parts (closures) produced by investment casting and after about 6 months of daily wear (Italy) are shown in ***Figure 17***. Like in numerous own R&D investment casting trials, ‘925 Ag New’ alloys are judged easy-to-cast with no relevant process modifications required if compared to standard sterling silver. Fluidity & reusability in casting of 925 AgNew has been ranked as at least comparable to standard 925 sterling silver. For sake of completeness, it should be mentioned that ‘925 Ag New’ alloys are also fire-stain free due to the absence or minimum amount Copper in the alloy, which is ranked as a positive side-effect by manufacturers. The comparably low as-cast hardness was noted and requires a single thermal treatment for age-hardening from the as-cast state. Age-hardening of the bracelet closure proved to provide efficient strength and stiffness to allow for secure fixing of the closures.

Figure 17: *Manufacturing with ‘925 Ag New’ alloy**,** investment cast ring and bracelet closure after ~ 6 months of wear (Italy)*

Continuous casting of wire and sheet material was performed successfully, followed by wire drawing and chain making as well as sheet forming, stamping and turning for wedding ring manufacture as well as watch case production.

Workability and work hardening properties consistently were ranked superior to 925 standard sterling silver by different manufacturers. Figures 18 to 21 show examples of manufactured parts before and after accelerated lab tarnish tests or real-life wear, respectively, confirming a high level of tarnish resistance.

Figure 18: *Manufactured chains of '925 Ag New' alloy**,** before (upper sample) and after accelerated lab tarnish test*

Figure 19: *Manufactured wedding bands of '925 Ag New' alloy**,** before (left sample) and after accelerated lab tarnish test*

Figure 20: *Manufactured watch cases of '925 Ag New' alloy compared to reference silver alloys after an accelerated lab tarnish test*

Figure 21: *Finalised watch of '925 Ag New' after 6 months of real-life wear (Italy)*

DISCUSSION AND CONCLUSIONS

Based on the metallurgical knowledge and insight into both tarnish phenomena and reliable tarnish testing which was gathered and shared over many years (see references), numerous new alloy recipies were studied for tarnish resistance by a combination of complementing acclerated lab tarnish test. An alloy composition range was identified, where Copper is at best fully replaced by other base metals like Indium, Tin and/or Zinc and where small additions of Palladium assist in an increase of tarnish resistance as well as in provision of age-hardening properties.

Based on lab analysis and confirmed by extensive real-life trials, these new 925 Ag alloys are characterised by a significantly increased tarnish resistance, equal to premium 9k gold alloys. Although such alloys can not be considered longterm tarnish-free, as clearly documented by real-life trials, this should allow its use in a natural way, i.e. without the need for plating processes, so that the fabricated jewellery also benefits from its true and extraordinary natural whiteness. Manufacturing trials using different production routes revealed overall working properties identical (casting) or superior (mechanical working) to standard sterling silver.

Obviously, even small additions of Palladium add significantly to the raw material costs of the newly developed alloys. However, this can be balanced (depending on the Pd amount and the fluctuating precious metal fixing) by cost savings due to avoidance of plating processes with pure Silver and especially Rhodium. As a side effect, nowadays gaining more and more importance, the avoidance of plating processes is ecologically favorable.

Given the fact, that such coatings anyway quickly wear off (partially) in real life, followed by non-uniform heavy tarnishing of e.g. an underlying standard sterling silver alloy base, the newly developed alloys potentially can lead to higher consumer satisfaction during the jewellery life-time. Few years after market launch in 2017, it remains challenging, however, to transfer the claimed benefits to jewellery manufacturers and consumers. Communication and marketing must be incisive but honest, always keeping in mind that tarnishing of 925 silver alloys can only be delayed, even if significantly as claimed for the newly developed alloys described by the present study, but it can never be completely avoided in all possible real-life scenarios.

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